Revision 2 (Amendment)
Quality Assurance Project Plan (QAPP)

Remedial Action/Groundwater Treatment
Waukegan Manufactured Gas and Coke Plant Site

Operable Unit 2 of the Outboard Marine Corporation Superfund Site
Waukegan, Illinois

Prepared December 2008

Burns & McDonnell
SINCE 1898

QUALITY ASSURANCE PROJECT PLAN (QAPP)

PROJECT TITLE: REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

REVISION NUMBER: 2 (AMENDMENT)

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[1] Notations reference applicable sections and corresponding elements from "Guidance for Quality Assurance Project Plans", EPA QA/G-5, December 2002.

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LIST OF ACRONYMS AND SHORT FORMS

BMcD	-	Burns & McDonnell
DQOs	-	Data Quality Objectives
EDDs	-	Electronic Data Deliverables
EMT	-	Environmental Monitoring and Technologies, Inc.
ESS	-	Environmental Sampling Supply, Inc.
FS	-	Feasibility Study
FSP	-	Field Sampling Plan
FSS	-	Field Support Section
GOU	-	Groundwater Operable Unit
GRZ	-	Groundwater Remediation Zone
GW RA	-	Groundwater Remedial Action
HAZWOPER	-	Hazardous Waste Operations and Emergency Response
ID	-	Identification
IDL	-	Instrument Detection Limit
IEPA	-	Illinois Environmental Protection Agency
LCS	-	Laboratory Control Sample
LIMS	-	Laboratory Information Management System
MDL	-	Method Detection Limit
MNA	-	Monitored Natural Attenuation
MS/DUP	-	Matrix Spike/Laboratory Duplicate
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
PARCCS	-	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
%R	-	Percent Recovery
QA	-	Quality Assurance
QA/QC	-	Quality Assurance/Quality Control
QAPP	-	Quality Assurance Project Plan
QC	-	Quality Control
QEC	-	Quality Environmental Containers, Inc.
PDF	-	Adobe Portable Document File
PPE	-	Personal Protective Equipment
RA	-	Remedial Action

LIST OF ACRONYMS AND SHORT FORMS (Continued)

RCRA	-	Resource Conservation and Recovery Act
RD	-	Remedial Design
RD SOW	-	Remedial Design Scope of Work
RDAOC	-	Remedial Design Administrative Order on Consent
RDWP	-	Remedial Design Work Plan
RI	-	Remedial Investigation
RI/FS	-	Remedial Investigation/Feasibility Study
ROD	-	Record of Decision
RPD	-	Relative percent Difference
Site	-	Waukegan Manufactured Gas and Coke Plant Site
SDG	-	Sample Delivery Group
SOPs	-	Standard Operating Procedures
SOW	-	Scope of Work
STAT	-	STAT Analysis Corporation
U.S. EPA	-	United States Environmental Protection Agency

1.0 INTRODUCTION

United States Environmental Protection Agency (U.S. EPA) policy requires that all work performed by or on behalf of U.S. EPA involving the collection of environmental data be implemented in accordance with a U.S. EPA-approved Quality Assurance Project Plan (QAPP). The QAPP is a planning document that provides a "blueprint" for obtaining the type and quantity of data needed to support environmental decision making. The QAPP integrates all technical and quality aspects of a project and documents all quality assurance (QA), quality control (QC), and technical activities and procedures associated with planning, implementing, and assessing environmental data collection operations.

Earlier versions of this QAPP were prepared by Conestoga-Rovers & Associates and this revision was prepared by Burns & McDonnell Engineering Company, Inc. (BMcD) in accordance with the U.S. EPA QAPP guidance documents "EPA Requirements for Quality Assurance Project Plans", EPA QA/R-5, March 2001 (Reissued May 2006), "EPA Guidance for Quality Assurance Project Plans", EPA QA/G-5, December 2002, and "Region 5 Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Revision 0", June 2000. In accordance with these documents, there are four basic groups of elements that must be included in a QAPP. These four groups and associated elements follow:

- Group A - Project Management. The elements in this group include all aspects of project management, project objectives, and project history.
- Group B - Data Generation and Acquisition. The elements in this group include descriptions of the design and implementation of all measurement systems that will be used during the project.
- Group C - Assessment/Oversight. The elements in this group encompass the procedures used to ensure proper implementation of the QAPP.
- Group D - Data Validation and Usability. The elements in this group cover the QA activities that occur after the data collection phase of the project is completed.

The elements that comprise project management, data generation and acquisition, assessment/oversight, and data validation and usability for the Remedial Action - Groundwater Operable Unit (RA-GOU) activities to be conducted at the Waukegan Manufactured Gas and Coke Plant Site (Site) in Waukegan, Illinois are documented in

this QAPP. This QAPP is Attachment E of the "Final Design Report, Groundwater Operable Unit, Waukegan Manufactured Gas and Coke Plant Site, Waukegan, Illinois". This QAPP will be modified as necessary to address additional studies and other work associated with the Groundwater Remedial Action (GW RA) at the Site. The modifications to this QAPP will be submitted with future work plans associated with the RA.

The GW RA consists of a combination of a short-term groundwater extraction and on-site treatment re-injection system (Phase 1 GW RA) and a long-term Monitored Natural Attenuation (MNA) remedy (Phase 2 GW RA).

2.0 PROJECT ORGANIZATION [2.1.4] A4[2]

The responsibilities of management, QA personnel, field personnel, and laboratory personnel are provided in the following subsections. Additionally, any special training/certification requirements for the project are identified and an organization chart that identifies the lines of communication among the participants in the RA activities is presented herein.

2.1 MANAGEMENT RESPONSIBILITIES [2.1.4] A4

The "Performing Respondents" have selected BMcD and Barr Engineering as the technical consultants for the RA activities at the Site. BMcD has overall technical responsibility for the Remedial Action - Groundwater Operable Unit groundwater data collection activities at the Site. BMcD's Project Manager is ultimately responsible for ensuring that the project objectives are achieved. BMcD's Project Manager has selected a project team consisting of BMcD's technical personnel (engineering, chemistry, and data management), QA personnel, Barr's technical personnel, and the analytical laboratories. BMcD's Project Manager for the RA activities and his specific responsibilities follow:

Eduardo Gasca, P.E. - Project Manager – BMcD

- technical representation for Performing Respondents;
- overview of field activities;
- overview of laboratory activities;
- advise on corrective actions;
- preparation and review of reports;
- coordinate BMcD's technical group;
- final evidence file custodian; and
- approval of the QAPP.

Each analytical laboratory's Project Manager is responsible for ensuring that the project objectives are achieved by the laboratory. The primary laboratory selected for this

[2] Notations reference applicable sections and corresponding elements from "Guidance for Quality Assurance Project Plans", EPA QA/G-5, December 2002.

project is Environmental Monitoring and Technologies, Inc. located at 8100 N. Austin Avenue in Morton Grove, Illinois (phone: 847-967-6666; fax: 847-967-6735).[3] The laboratory's Project Manager and her specific responsibilities follow:

Arminta Priddy - Project Manager - EMT

- ensure all resources of the laboratory are available on an as-required basis;
- review of final analytical reports; and
- approve final reports prior to submission to BMcD.

The secondary (back-up) laboratory selected for this project is STAT Analysis Corporation located at 2242 W. Harrison Street, Suite 200, Chicago, Illinois (phone: 312-733-0551; fax: 312-733-2386). The laboratory's Project Manager and his specific responsibilities follow:

Pinaki Banerjee – Project Manager & QA/QC Director – STAT

- ensure all resources of the laboratory are available on an as-required basis;
- review of final analytical reports; and
- approve final reports prior to submission to BMcD.

The U.S. EPA Region 5 Remedial Project Manager is responsible for overview of this project. He also is responsible for submitting this QAPP and any subsequent revisions or amendments to the appropriate U.S. EPA personnel for review and approval and for providing approval of the QAPP. Kevin Adler is the Remedial Project Manager for the RA at the Site.

The Illinois Environmental Protection Agency (IEPA) representative is responsible for reviewing and providing comments to U.S. EPA on project plans for the RA activities. Erin Rednour is IEPA's representative for this project.

[3] Responsibilities of the primary and secondary (back-up) laboratory are discussed in Section 2.4 below.

2.2 QUALITY ASSURANCE RESPONSIBILITIES [2.1.4] A4

Project team members with QA responsibilities include BMcD's QA Officer, BMcD's Field QA Officer, and the laboratory's QA Officer. These individuals and their specific responsibilities follow:

Sharon Shelton - Quality Assurance Officer - BMcD

- overview and review field QA/QC;
- review laboratory QA/QC;
- supervise and review performance of data validation and assessment;
- advise on laboratory corrective action procedures;
- supervise and review QA report preparation;
- QA/QC representation of project activities; and
- approval of QAPP.

Tim Gilles - Field Quality Assurance Officer - BMcD

- management of field activities and field QA/QC;
- field data assessment;
- internal field technical system audits;
- technical representation of field activities;
- preparation of standard operating procedures (SOPs) for field activities;
- implement and document field corrective actions, if necessary; and
- approval of QAPP.

Brian Goyette - QA Officer - EMT
Pinaki Banerjee - QA Officer - STAT

- coordinate and overview of laboratory systems audits;
- overview of QA/QC documentation;
- conduct detailed data review;
- implement and document laboratory corrective actions, if required;
- technical representation of laboratory QA procedures;

- oversee preparation of laboratory SOPs; and
- approval of the QAPP.

The U.S. EPA Region 5 Field Support Section (FSS) Quality Assurance Reviewer is responsible for reviewing and providing final approval of the QAPP.

2.3 FIELD RESPONSIBILITIES [2.1.4] A4

BMcD and Barr Engineering will conduct all field sampling related to the RA activities. The specific procedures for field sample collection are presented in the Field Sampling Plan (FSP), which is Attachment D of the Final Design Report, Groundwater Operable Unit Submittal. Surveying of cell locations will be performed by an Illinois-licensed surveyor consistent with the requirements of the FSP.

BMcD's field sampling team will consist of technical staff from BMcD's Downers Grove, Illinois office. Barr's sampling teams will be comprised of technical staff from Barr's Minneapolis, Minnesota office. BMcD's Field QA Officer (Tim Gilles) will have overall responsibility for documenting any non-conformances and subsequent corrective actions. The Field QA Officer or any field team member can identify and report non-conformances.

2.4 LABORATORY RESPONSIBILITIES [2.1.4] A4

EMT will be the primary laboratory for this project and will perform all analyses of samples collected during the Site activities. STAT, the secondary laboratory, will provide back-up analytical services. Groundwater samples collected to monitor the groundwater extraction and treatment operation will be analyzed for ammonia, arsenic, and phenolics.

The secondary laboratory may be used for QA/QC purposes to assess the performance of the primary laboratory.

The specific responsibilities of laboratory personnel involved in the project follow:

Mary Lubitov - Wet Chemistry Supervisor - EMT
Richard Baker - Operations Manager - STAT

- coordinate laboratory analyses;
- supervise in-house chain-of-custody;
- schedule sample analyses;
- oversee data review; and
- oversee preparation of analytical reports.

Martina Hanson - Sample Custodian - EMT
Chris Forst - Sample Custodian - STAT

- receive and inspect the incoming sample containers;
- record the condition of the incoming sample containers;
- sign appropriate documents;
- verify correctness of chain-of-custody documentation;
- notify project manager of any non-conformances identified during sample receipt and inspection;
- assign a unique identification number and customer number, and enter each into the sample receiving log;
- initiate transfer of the samples to appropriate lab sections; and
- control and monitor access/storage of samples and extracts.

2.5 SPECIAL TRAINING/CERTIFICATION REQUIREMENTS [2.1.4] A4

BMcD and Barr field sampling team members are required to have received the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) safety training and annual 8-hour refresher courses required by 29 CFR Parts 1910 and 1926. On-site subcontractor personnel involved in invasive activities (e.g., excavation) are required to have received the same training. The subcontractor is responsible for compliance of their personnel with the applicable regulations.

Laboratory personnel training records are maintained at the laboratory. No special training or certification requirements are required for the laboratory for this project.

2.6 PROJECT ORGANIZATION [2.1.4] A4

Figure 2.1 presents the organizational chart for the RA activities.

3.0 PROBLEM DEFINITION/BACKGROUND INFORMATION [2.1.5] A5

The purpose of the Remedial Action - Groundwater Operable Unit and background information for the Site are presented in the following sections.

3.1 PROBLEM DEFINITION [2.1.5] A5

The purpose of the groundwater pump and treat system is to extract contaminated groundwater from the bottom of the aquifer within the Groundwater Remediation Zone (GRZ) , reduce the mass of ammonia, arsenic, and phenols through on-site treatment , and return clean groundwater back to the ground. It is expected that an 80% reduction in the mass of ammonia, arsenic, and phenols will permit the Phase 2 GW RA Monitored Natural Attenuation to be successful. The test to determine compliance with the 80% reduction is described in the Performance Standard Verification Plan, Attachment C to the Final Design Report.

3.2 BACKGROUND INFORMATION [2.1.5] A5

The Site background is provided in Section I and II of the Record of Decision, September 1999.

3.2.1 SITE DESCRIPTION AND HISTORY [2.1.5] A5

The Site occupies 36 acres in Waukegan, Illinois on a peninsula separating Waukegan Harbor on the west from Lake Michigan on the east. Commercial and industrial land and a harbor surround the Site on the north, west, and south. To the east of the Site lies Waukegan Beach recreational area. The Site was initially purchased and developed as a creosote wood-treating plant in 1908. The creosote plant was dismantled sometime after 1917. The Site was then developed as a large manufactured gas and coke plant and operated under various owners until 1969. The coke plant structures were subsequently demolished in 1972 and the Site was filled and leveled. Between 1973 and 1989 the Site was used for various activities including fire training, public parking, and snowmobile

testing. The northwest portion of the Site is currently used for seasonal boat and trailer storage.

The Site is presently owned by the City of Waukegan, Illinois.

3.2.2 PREVIOUS INVESTIGATIONS AND RESPONSE ACTIONS [2.1.5] A5

Results of previous investigations and response actions are provided in the Remedial Investigation (RI) Report, February 1995, the Feasibility Study (FS) Report, November 1998, the Pilot Project, July 2001, the Groundwater Plume Delineation Report, December 2001, and the Nitrification Study, May 2004.

3.2.3 PAST DATA COLLECTION ACTIVITIES [2.1.5] A5

A Remedial Investigation/Feasibility Study (RI/FS) was conducted at the Site between 1992 and 1998. Results of the RI/FS data collection activities identified coal tar compounds, arsenic, ammonia, phenols and several other compounds as chemicals of concern at the Site.

A Pilot Project was completed in 2000/2001. The results are reported in the Pilot Project Report July 2001. The Pilot Project concluded that the principle of cell based low flow extraction and re-injection would work. Groundwater Plume Delineation Report, December 2001 updated the GRZ from the RI and Record of Decision (ROD). The Nitrification Study, May 2004 demonstrated that biological sequencing batch reactors would work as the primary treatment technology.

3.2.4 CURRENT STATUS [2.1.5] A5

A Remedial Design Work Plan (RDWP) for the Site was prepared and submitted to U.S. EPA in August 2001 and was subsequently approved in December 2001. The RDWP was prepared consistent with the Remedial Design Scope of Work (RD SOW), which is Attachment II to the Administrative Order on Consent for Remedial Design

(RDAOC) at the Waukegan Coke Plan Site, U.S. EPA Docket No. V-W-01-C-651. The GW RA is a component of the RDWP.

3.3 PROJECT/TASK DESCRIPTION [2.1.6] A6

The scope of work to be completed during the groundwater remedial action consists of the following tasks:

i) install approximately 500 groundwater extraction and re-injection wells up to 30 feet deep;

ii) install extraction and re-injection water piping;

iii) install forcemains and control lines to water treatment plant;

iv) renovate existing building;

v) install groundwater treatment plant in renovated building;

vi) operate groundwater extraction, treatment and re-injection for up to 5 years; and

vii) collect samples of extracted water, re-injection water, and monitoring well water and analyze for ammonia, arsenic, and phenols.

This QAPP is applicable to Task vii) above. Task vii) is described in the following subsection of this QAPP.

This QAPP applies to the Phase 1 GW RA.

The groundwater that is the target of the remediation lies in the lower portion of the upper (sand) aquifer within an area known as the GRZ. The GRZ is presented on Figure 3.1 of the Final Design Report, Groundwater Operable Unit. Groundwater will be extracted, treated, and re-injected. Extraction and re-injection will occur in a pattern of cells over the GRZ. A typical cell will be 110 feet long by 100 feet wide. Each cell will have one line of 5 or 6 extraction wells backfilled by 2 lines of five re-injection wells for a total of 15 or 16 wells per cell. There are 36 cells. Cells are expected to run for approximately 63 days each. Normally 3 cells will be operating simultaneously except during startup and shutdown.

The performance standard for the Phase 1 GW RA is an 80% reduction in ammonia, arsenic, and phenols in the bottom 5 feet of the aquifer. Ammonia, arsenic, and phenols are the data that will be used for environmental decision making and therefore, they are the subject of this QAPP.

3.3.1 GROUNDWATER MONITORING [2.1.6] A6

The ROD performance goal for the Phase 1 GW RA is an 80% reduction in the mass of ammonia, arsenic, and phenols in the bottom 5 feet of the sand aquifer. Consequently, ammonia, arsenic, and phenols are the only parameters that will be used for environmental decision making. As a further result, ammonia, arsenic, and phenols in water are the only parameters covered in this QAPP. Table 3.1 provides a summary of the sampling and analysis program. Table 3.2 provides the targeted quantitation limits.

3.3.1.1 EXTRACTED GROUNDWATER [2.1.6] A6

Initial Monitoring

Initial Monitoring will be conducted to define a baseline condition on each new cell that is brought online. During the first three days of cell operation, a composite sample consisting of equal parts from each extraction well will be collected each day. The volume of each aliquot will be the sample container volume divided by the number of wells that will be part of the composite sample.

The initial concentration for each cell (Co) will be the maximum concentration measured in the 3 samples. Initial Monitoring procedures may be modified with experience.

Performance Monitoring

Performance Monitoring is weekly monitoring of the extraction wells beginning when 2/3 of a cell water volume has been removed. This would normally be expected to occur after 6 weeks unless there has been an interruption in operations. If the extraction re-injection proceeds as expected each cell will have 3 sets of performance monitoring

samples collected once per week for the last three weeks of cell operation. Each sample will be a composite of equal parts of each extraction well in the cell.

Informational Monitoring

Additional parameters will be monitored approximately every 3 weeks to provide a broader array of parameters to help operators optimize the removal of ammonia, arsenic, and phenols. However, as these parameters may not be used in environmental decision making with respect to achieving performance goals, these parameters are not included in this QAPP.

Detailed Monitoring One Cell

One cell will be selected for more intensive monitoring after the extraction, treatment, re-injection has reached a smooth normal operation. The more intense monitoring will be used to optimize normal monitoring and to refine the groundwater model used to predict the aquifer response to the Phase 1 GW RA.

Cell 4 has been tentatively selected for Detailed Monitoring assuming startup has been successful in Cells 1 through 3. Two monitoring well nests will be installed within Cell 4. Four individual wells will be set at each location. The two feet long well screens will be set above the till at 0-2 feet, 3-5 feet, 8.5-10.5 feet, and 14-16 feet.

The monitoring wells will be sampled once after well development and twice weekly when Cell 4 begins operation. The extraction wells will be monitored individually each day for the first 5 days of cell operation and then a composite sample will be collected weekly at the same time as one of the monitoring well sample events.

3.3.1.2 RE-INJECTION WATER [2.1.6] A6

Re-injection water will be sampled in the Water Treatment Plant Effluent Holding Tank on a weekly basis.

3.4 PROJECT SCHEDULE [2.1.6] A6

The Phase 1 GW RA groundwater treatment plant construction was completed in August 2008 and deemed complete on October 10, 2008. The operation of the groundwater treatment system started in September 2008 and is expected to treat extracted groundwater from 36 extraction/re-injection cells at a rate of one cell every 63 to 70 days once the groundwater treatment plant is capable of treating the average flow of 35 gallons per minute. It is projected that the remediation of the Phase 1 groundwater component of the RA will be accomplished within 3 to 5 years from the start of the operations.

4.0 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA [2.1.7]

The data quality objectives and measurement performance criteria for the GW RA activities are presented in the following subsections.

4.1 DATA QUALITY OBJECTIVES [2.1.7] A7

Data quality objectives (DQOs) are qualitative and quantitative statements derived from the outputs of each step of the DQO process. The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application.

There are seven steps in the DQO process that include:

1. stating the problem;
2. identifying the decision;
3. identifying inputs to the decision;
4. defining the boundaries of the study;
5. developing a decision rule;
6. specifying limits on decision errors; and
7. optimizing the design for obtaining data.

The details of DQO process for the groundwater sampling program are provided below.

The problem as identified in Section 3.1 of this QAPP is to confirm that 80% of ammonia, arsenic, and phenols have been removed from the bottom 5 feet of the aquifer within the GRZ.

Calculating 80% removal of ammonia, arsenic, and phenols requires data of known quality and accuracy for ammonia, arsenic, and phenols.

4.2 MEASUREMENT PERFORMANCE CRITERIA [2.1.7] A7

The measurement performance criteria for precision, accuracy, representativeness, completeness, and comparability are provided in the following subsections.

4.2.1 PRECISION [2.1.7] A7

Precision is a measure of the degree to which two or more measurements of the same characteristic (i.e., analyte, parameter) under the same or similar conditions are in agreement.

4.2.1.1 FIELD PRECISION CRITERIA [2.1.7] A7

Precision of the field sample collection procedures will be assessed by the data from analysis of field duplicate samples. Relative percent differences (RPDs) will be calculated for detected analytes from field duplicate sample sets. Field duplicate samples will be collected at a minimum frequency of 1 per 20 verification samples. The equation to be used to determine precision is presented in Section 7.3 of this QAPP. An RPD of 35 percent for groundwater sample field duplicates will be used as an advisory limit. Professional judgment will be used for any data qualification.

4.2.1.2 LABORATORY PRECISION CRITERIA [2.1.7] A7

Laboratory precision will be assessed through the calculation of RPDs for replicate/duplicate sample analyses. In general, these will be matrix spike/matrix spike duplicate (MS/MSD) samples collected at a minimum frequency of 1 per 20 samples. The equation to be used to determine precision is presented in Section 7.3 of this QAPP. Precision control limits for the analyses are presented in Table 4.1.

4.2.2 ACCURACY [2.1.7] A7

Accuracy is the extent of agreement between an observed value (i.e., sample result) and the accepted or true value for the parameter being measured.

4.2.2.1 FIELD ACCURACY CRITERIA [2.1.7] A7

The criteria for accuracy of the field sample collection procedures will be to ensure that samples are not affected by sources external to the sample, such as sample contamination by ambient conditions or inadequate equipment decontamination procedures. Field sampling accuracy will be assessed by the data from field blank samples.

Rinsate blank samples will be collected at a frequency of one per twenty sampling equipment decontamination procedures or a least once per day of sampling equipment cleanings, whichever is more frequent. Rinsate blank samples will be collected by routing laboratory-provided deionized water through decontaminated sampling equipment. Rinsate blank samples will be analyzed to check procedural contamination and/or ambient conditions and/or sample container contamination at the Site that may cause sample contamination. Rinsate blank samples will not be collected for waste characterization sampling and for samples collected using pre-cleaned or pre-cleaned, disposable sampling equipment.

Rinsate blank samples should not contain target analytes. The rinsate blank sample data will be evaluated using the procedures specified in Section 7.3 of this QAPP. Accuracy will be ensured by adhering to all sample handling procedures, sample preservation requirements, and holding time periods.

4.2.2.2 LABORATORY ACCURACY CRITERIA [2.1.7] A7

Laboratory accuracy will be assessed by determining percent recoveries from the analysis of laboratory control samples (LCSs) or standard reference materials. Accuracy relative to the sample matrix will be assessed by determining percent recoveries from the analysis of MS/MSD samples. MS/MSD samples will be collected and designated for analyses at a minimum frequency of 1 per 20 or fewer samples. The equation to be used to determine accuracy for this project is presented in Section 7.3 of this QAPP. Accuracy control limits are presented in Table 4.1.

4.2.3 REPRESENTATIVENESS [2.1.7] A7

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental condition of a site. Representativeness also reflects the ability of the sample team to collect samples and laboratory personnel to analyze those samples in such a manner that the data generated accurately and precisely reflect the conditions at a site.

4.2.3.1 FIELD REPRESENTATIVENESS CRITERIA [2.1.7] A7

Representativeness is dependent upon the proper design of the sampling program. The representativeness criteria for field sampling will be to ensure that the sampling grids are properly established at the site, that the correct monitoring wells are sampled, and that the sampling procedures in the FSP are followed. The sampling programs were designed to provide data representative of Site conditions. During development of these programs, consideration was given to the agreed definition of the GRZ, existing analytical data, physical setting and processes, and constraints inherent to the Superfund program. The rationale for the sampling network is provided in Section 3.3 of this QAPP.

4.2.3.2 LABORATORY REPRESENTATIVENESS CRITERIA [2.1.7] A7

The representativeness criteria for laboratory data will be to ensure that the proper analytical procedures are used for sample preparation , sample analysis, and that sample holding times are met. Additionally, the accuracy and precision of the laboratory data affect representativeness. The laboratory representativeness criteria will include achieving the accuracy and precision criteria for the sample analyses.

4.2.4 COMPARABILITY [2.1.7] A7

Comparability is an expression of the confidence with which one data set can be compared with another.

4.2.4.1 FIELD COMPARABILITY CRITERIA [2.1.7] A7

The criteria for field comparability will be to ensure and document that the sampling networks designed for the RD activities are properly implemented and the sampling procedures in the FSP are followed for the duration of the sampling programs.

4.2.4.2 LABORATORY COMPARABILITY CRITERIA [2.1.7] A7

The criteria for laboratory data comparability will be to ensure that the analytical methods used for the RA sampling and analysis events are comparable to the methods used for previous sampling events. The analytical methods identified in Section 5.3 of this QAPP are comparable to the methods used to generate data for previous investigations.

4.2.5 COMPLETENESS [2.1.7] A7

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

4.2.5.1 FIELD COMPLETENESS CRITERIA [2.1.7] A7

No field measurements are used in environmental decision making.

4.2.5.2 LABORATORY COMPLETENESS CRITERIA [2.1.7] A7

The criteria for laboratory completeness will be that a minimum of 90 percent of the laboratory data will be determined to be valid (usable) for the intended purpose. The procedure for determining laboratory data validity is provided in Section 7.0 of this QAPP. The equation for calculating completeness is presented in Section 7.3.4 of this QAPP.

4.2.6 SENSITIVITY [2.1.7] A7

Sensitivity is the ability of a method or instrument to detect a parameter to be measured at a level of interest.

4.2.6.1 FIELD SENSITIVITY CRITERIA [2.1.7] A7

The field measurements for the project only consist of distance measurements. Sensitivity is not applicable to these measurements.

4.2.6.2 LABORATORY SENSITIVITY CRITERIA [2.1.7] A7

The sensitivity requirements for the laboratory analyses are provided in Table 3.2. Where evaluation criteria for a sampling program exist, the concentrations of the evaluation criteria are included in the table. The analytical methods are sufficiently sensitive for the project.

4.3 SPECIAL TRAINING/CERTIFICATION REQUIREMENTS [2.1.8] A8

Special training/certification requirements for this project were provided in Section 2.5.

4.4 DOCUMENTATION AND RECORDS [2.1.9] A9

The documents, records, and reports generated during the RA are identified in the following subsections.

4.4.1 FIELD AND LABORATORY RECORDS [2.1.9] A9

Documents and records generated during the project include sample collection records, QC sample records, laboratory records, and data handling records. A brief description

of these documents and records are provided below. Detailed information on these records is provided in subsequent sections of this QAPP.

Sample collection records that will be used during the sampling activities include field logbooks, stratigraphic logs, chain-of-custody records, and shipping papers.

QC sample records that will be used during the project to document the generation of QC samples include field logbooks for recording rinsate blank samples, field duplicate samples, and MS/MSD samples. The laboratory will maintain appropriate documentation of trip blank sample preparation, quality records for deionized water sent for rinsate blank samples, and sample integrity information. Records of sample preservation will be maintained in field logbooks and by the laboratory.

Laboratory records that will be maintained for the project include sample receipt documentation, field and laboratory chain-of-custody documentation, sample container cleanliness certifications, reagent and standard reference material certifications, sample preparation records, sample analysis records (e.g., run logs), instrument/raw data, QC data, calibration data, corrective action reports, and final reports.

Data handling records that will be maintained include verification of computer programs used to manipulate or reduce raw data into final results and data validation reports. The laboratory will maintain documentation of data verification and reduction procedures, as necessary, for the analyses used during the RA activities. BMcD will maintain checklists, notes, and reports generated during the external data validation process.

4.4.2 DATA REPORTING FORMAT [2.1.9] A9

Field data will be recorded in bound logbooks or on standard forms (e.g., chain-of-custody logs). The details for recording field data are provided in Section 5.2.2.1 of this QAPP. Field data will be primarily generated from observations. These data will be tabulated and included in project reports or submittals, as appropriate.

Laboratory reports for the RA activities will consist of the following data deliverables:

1. Case Narrative
   - i) date of issuance;
   - ii) any deviations from intended analytical strategy;
   - iii) laboratory batch number;
   - iv) number of samples and respective matrices;
   - v) project name and number;
   - vi) condition of samples "as received";
   - vii) discussion of whether or not sample holding times were met;
   - viii) discussion of technical problems or other observations which may have created analytical difficulties; and
   - ix) discussion of any laboratory quality control checks which failed to meet project criteria.
2. Chemistry Data Package
   - i) dates of sample collection, receipt, preparation, and analysis;
   - ii) cross-reference of laboratory to project sample identification numbers;
   - iii) description of data qualifiers used;
   - iv) methods of sample preparation and analysis;
   - v) sample results in tabular format;
   - vi) MS/MSD data, LCS data, method blank data, ; and
   - vii) fully executed chain-of-custody document.

Raw instrument data (including calibration data and instrument performance checks), method detection limit (MDL) studies, instrument detection limit (IDL) studies, and method performance and validation studies will be maintained by the laboratory.

5.0 DATA GENERATION AND ACQUISITION [2.2]

The design and implementation of the measurement systems that will be used during the RA activities, including sampling procedures, analytical procedures, and data handling and documentation are detailed in the following subsections.

5.1 SAMPLING PROCESS DESIGN [2.2.1] B1

The rationale for the sampling programs is provided in Attachment B of the Final Design Report and was detailed in Section 3.3 of this QAPP. The sampling programs were developed based on the requirements of the SOW and refined through planning meetings.

5.1.1 SAMPLING METHODS [2.2.2] B2

Sampling methods for the collection of groundwater samples are provided in the FSP and Table 5.2.

5.1.2 FIELD EQUIPMENT AND SAMPLE CONTAINER CLEANING PROCEDURES [2.2.2] B2

Equipment cleaning/decontamination procedures are provided in section 2.2.2 of the FSP. Sample containers will be provided by the laboratory performing the sample analyses. EMT's vendors for sample containers are Quality Environmental Containers (QEC), Inc. of Beaver, West Virginia and Environmental Sampling Supply (ESS) of Oakland, California . STAT's vendor for sample containers is QEC. All containers will be pre-cleaned in accordance with the U.S. EPA guidance document entitled "Specifications and Guidance for Contaminant-Free Sample Containers", EPA 540/R-93/051. Certificates of analysis for each lot and type of container will be maintained by the laboratory. Example Certificates of analysis are presented as Figures 5.1 and 5.2.

5.1.3 FIELD EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION REQUIREMENTS [2.2.2] B2

Field (sample collection) equipment will be inspected and tested prior to being shipped to the field. Maintenance logs for all field equipment owned the Site or by BMcD are kept in BMcD's field equipment logs at the BMcD Downers Grove office. Maintenance records for any rented equipment are kept by the equipment supplier. Prior to use in the field, the equipment is checked again, generally during field calibration, and the performance information is recorded in the field logbook. All equipment shipped back from the field is inspected and tested upon return. Any required maintenance is performed and documented prior to the equipment being returned to service.

Critical spare parts for field equipment and replacement field equipment are available at the Site or at the BMcD Downers Grove office and can be shipped for overnight delivery, picked up at the BMcD office, or delivered to the field when the need is identified. Alternately, field equipment vendors (e.g., Ashtead Technology Rentals) can provide replacement equipment if needed. The replacement equipment can be shipped for overnight delivery as necessary.

5.1.4 INSPECTION AND ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND SAMPLE CONTAINERS [2.2.2] B2

The field supplies for the RA activities consist of detergent (Alconox) for equipment cleaning, distilled water for sample collection equipment rinsing, deionized water for final sample collection equipment rinsing and for collecting field (equipment rinsate) blank samples, and sample containers to collect the samples. Alconox, which is a standard laboratory-grade detergent, is obtained from USA BlueBook. Distilled water will be purchased as needed from a variety of vendors. Deionized water is obtained from USA BlueBook or Hach Company.

Sample containers will be provided by the laboratory performing the analyses, which will maintain documentation of the purity/cleanliness for these materials. The laboratory's QA Officer is ultimately responsible for ensuring that these materials are acceptable for the project. The acceptability of these materials for use will be evaluated by reviewing lot

analysis certificates. Water and containers that do not meet the laboratory's acceptability requirements will not be shipped to the field.

5.2 SAMPLE HANDLING AND CUSTODY REQUIREMENTS [2.2.3] B3

The procedures for sample handling, labeling, shipping, and chain-of-custody documentation are provided in the subsections that follow.

5.2.1 SAMPLE HANDLING [2.2.3] B3

The procedures used to collect the samples are provided in the FSP. The samples collected during the groundwater RA will be analyzed for ammonia, arsenic, and phenols. Specifics of the sampling and analysis program are described in Section 3.3 of this QAPP. A summary of the sampling and analysis program is presented in Table 3.1. The containers for groundwater will be filled in the following sequence: ammonia, phenols, and finally arsenic. Table 5.1 identifies the requirements for the number of containers, container volume, container type (material of construction), preservation, holding time periods, packaging, and shipping for the analyses.

The sample numbering system for the Phase 1 GW RA was designed to uniquely identify each groundwater performance evaluation sample collected for analysis of ammonia, arsenic and total phenolics. For samples from individual sampling points, this numbering system consists of the sample location identification (ID), sample collection date, and sequential number beginning with 001 for each sample collected at that sampling location in any one calendar day.

Sample locations include extraction wells (E), the water treatment plant effluent holding tank sample port (SP), and monitoring wells (M).

EXAMPLE:
E-01-2-081007-001

Where: E = Extraction well
01 = Remediation cell number

2 = Extraction well 1 within remediation cell
081007 = Sampling date (YY/MM/DD)
001 = First sample collected on October 7, 2008.

For composite samples from a groundwater remediation cell, the sample numbering system is as follows: Sample location ID, remediation cell number, and sampling date remain the same. Each sample aliquot (portion collected from each well) is assigned an alphabetic identifier rather than a numeric sequence identifier.

EXAMPLE
E-01-081014-A
E-01-081014-B
E-01-081014-C
E-01-081014-D
E-01-081014-E

Where: E = Extraction well
01 = Remediation cell number
081014 = Sample date (YYMMDD)
A, B, C, D, E = sample aliquot (one aliquot collected from each active extraction well

The laboratory composites the aliquots for analysis and identifies the single composite sample as E-01-081014-A-E.

Field duplicate samples, rinsate blanks and trip blanks will be numbered using the following identifiers to avoid laboratory bias of field QC samples:

Duplicate sample = DUP-YYMMDD-XXX
Rinsate blank = BLANK-YYMMDD-XXX
Trip Blank = TRIP-YYMMDD-XXX

Samples designated for MS/MSD analysis will be identified as such in the remarks column of the chain-of-custody form.

Samples will be placed in shipping coolers containing bagged, cubed ice immediately following collection or placed in the field laboratory refrigerator located in a secured area of the GW treatment plant building. The samples will be grouped in the shipping cooler by the order in which the samples are collected, and shipped to the laboratory via an overnight courier service, generally on the day they are collected. The only exceptions to this procedure will be for samples collected after the courier service has picked up the shipment for the day (generally only at remote sites) and samples collected on a Sunday or holiday. In these instances, the samples will be shipped on the next business day. An example shipping form is provided in Attachment 2.

The laboratory will group all samples received each day into one sample delivery group (SDG).

5.2.2 SAMPLE CUSTODY [2.2.3] B3

Chain-of-custody is the sequence of possession of an item. An item (such as a sample or final evidence file) is considered to be in custody if the item is in actual possession of a person, the item is in the view of the person after being in his/her actual possession, or the item was in a person's physical possession but was placed in a secure area by that person. Field, laboratory, and final evidence files custody procedures are described in the subsections that follow.

5.2.2.1 FIELD CUSTODY PROCEDURES [2.2.3] B3

Logbooks will used to record field data collection activities. An example field logbook entry is presented in Figure 5.2. Entries into field logbooks will be described in as much detail as possible to ensure that a particular situation could be reconstructed solely from logbook entries. Field logbooks will be bound field survey books or notebooks with consecutively numbered pages. Logbooks will be assigned to field personnel and will be stored at the site field office when not in use. Each logbook will be identified by the project number (49452) and sequentially numbered by dates of use.

The title page of each logbook will contain the following information:

- BMcD contact information;
- logbook number;
- project name;
- project start date; and
- end date.

Entries into the logbook will contain a variety of information. At the beginning of each day's logbook entry, the date, start time, weather, names of all sampling team members present, and the signature of the person making the entry will be entered. The names of individuals visiting the site or field sampling team and the purpose of their visit will also be recorded in the field logbook.

All field measurements obtained and samples collected will be recorded. All logbook entries will be made in ink, signed, and dated with no erasures. If an incorrect logbook entry is made, the incorrect information will be crossed out with a single strike mark which is initialed and dated by the person making the erroneous entry. The correct information will be entered into the logbook adjacent to the original entry.

Whenever a sample is collected or a measurement is made, a detailed description of the location will be recorded in the logbook. Photographs taken at a location, if any, will also be noted in the logbook. All equipment used to obtain field measurements will be recorded in the field logbook. In addition, the calibration data for all field measurement equipment will be recorded in the field logbook or on standard field forms.

Samples will be collected following the sampling procedures documented in the FSP. The equipment used to collect samples, time of sample collection, sample description, volume and number of containers, and preservatives added (if applicable) will be recorded in the field logbook. Each sample will be uniquely identified using the sample numbering system provided in Section 5.2.1 of this QAPP.

The sample packaging and shipping procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred to another person or the laboratory. As few people as possible will handle the samples.
2. All sample containers will be identified by using sample labels that include the date of collection and analyses to be performed. Sample labels will be completed for each sample using waterproof ink. An example sample label is provided in Attachment 2.
3. Samples will be accompanied by a properly completed chain-of-custody form. The sample identification numbers and required analyses will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign and record the date and time on the form. The chain-of-custody form documents sample custody transfers from the sampler to another person, to the laboratory, or to/from a secure storage area.
4. Samples will be properly packaged for shipment (see Table 5.1) and dispatched to the laboratory for analysis with a separate signed chain-of-custody form enclosed in and secured to the inside top of each shipping cooler. Shipping coolers will be secured with custody seals for shipment to the laboratory. The custody seals are then covered with clear plastic tape to prevent accidental damage to the custody tape. An example of the custody seal to be used for this project is provided in Attachment 2.
5. If samples are co-located with a government agency or other entity, it is the responsibility of that entity to prepare its own chain-of-custody form for the samples. Information regarding the identity of the entity and the samples that are being co-located will be recorded in the field logbook.
6. All sample shipments will be accompanied by the chain-of-custody form identifying its contents. The chain-of-custody form is a two-part, carbonless-copy form. The form is completed by the sampling team which, after signing and relinquishing custody to the laboratory's courier, retains the bottom (yellow) copy. The laboratory's courier signs and relinquishes custody to the laboratory's sample custodian. The laboratory retains the fully executed original chain-of-custody form. An Adobe Portable Document File (PDF) copy of the form is included as part of the

data deliverables package. An example chain-of-custody form is provided in Attachment 2.

7. If the samples are sent by common carrier, a bill of lading (e.g., FedEx air bill) will be used and copies will be retained as permanent documentation. Commercial carriers are not required to sign the chain-of-custody form as long as the form is sealed inside the sample cooler and the custody tape remains intact.

5.2.2.2 LABORATORY CUSTODY PROCEDURES [2.2.3] B3

Laboratory sample custody begins when the samples are received at the laboratory. The laboratory's sample custodian will assign a unique laboratory sample identification number to each incoming sample. The field sample identification numbers, laboratory sample identification numbers, date and time of sample collection, date and time of sample receipt, and requested analyses will be entered into the sample receiving log. The laboratory's sample log-in, custody, and document control procedures are detailed in the appropriate SOPs in Attachment 1.

Following log-in, all samples will be stored within an access-controlled location and will be maintained properly preserved (as defined in Table 5.1) until completion of all laboratory analyses. Unused sample aliquots and sample extracts/digestates/distillates will be maintained properly preserved for a minimum of 30 days following receipt of the final report by BMcD. The lab will be responsible for the disposal of unused sample aliquots, sample containers, and sample extracts/digestates/distillates in accordance with all applicable local, state, and federal regulations.

The laboratory will be responsible for maintaining analytical log books and laboratory data. Raw laboratory data files will be inventoried and maintained by the laboratory for a period of five years, at which time BMcD will advise the laboratory regarding the need for additional storage.

5.2.2.3 FINAL EVIDENCE FILES CUSTODY PROCEDURES [2.2.3] B3

The final evidence file for the project will be maintained by BMcD and will consist of the following:

1. project plan;
2. project log books;
3. field data records;
4. sample identification documents;
5. chain-of-custody records;
6. correspondence;
7. references, literature;
8. final data packages;
9. miscellaneous - photos, maps, drawings, etc.; and
10. final report.

The final evidence file materials will be the responsibility of the evidentiary file custodian (BMcD's Project Manager) with respect to maintenance and document removal.

5.3 ANALYTICAL METHOD REQUIREMENTS [2.2.4] B4

The field and laboratory analytical methods that will be used during the RA activities are detailed in the following subsections.

5.3.1 FIELD ANALYTICAL METHODS [2.2.4] B4

There are no field analytical methods for the RA.

5.3.2 LABORATORY ANALYTICAL METHODS [2.2.4] B4

Groundwater samples will be analyzed off Site by the project laboratory.

The analytical methods that will be used by the laboratory for analyzing groundwater samples are presented in Table 5.2. SOPs for the analytical methods are presented in

Attachment 1. Method validation and detection limit study information for the analyses are included in the laboratories' SOPs.

The quantities and types of QC samples for the GW RA are included in Table 3.1.

5.4 QUALITY CONTROL REQUIREMENTS [2.2.5] B5

The field and laboratory QC requirements for the RA activities are discussed in the following subsections. Specific QC checks and acceptance criteria are provided in the SOPs in Attachment 1.

5.4.1 FIELD SAMPLING QUALITY CONTROL [2.2.5] B5

Field QC samples for this project include rinsate blank samples to determine the existence and magnitude of sample contamination resulting from ambient conditions or sampling procedures and field duplicate samples to assess the overall precision of the sampling and analysis event. The frequency of collection of these field QC samples was provided in Section 4.2 of this QAPP. The evaluation of field QC data is provided in Section 7.3 of this QAPP.

5.4.2 ANALYTICAL QUALITY CONTROL [2.2.5] B5

The laboratory QC requirements for the arsenic analyses to be performed for the GW RA include analyzing preparation blanks, initial calibration blanks, continuing calibration blanks, initial calibration verification standards, continuing calibration verification standards, interference check standards, MS/MSD samples, and LCSs. The analysis frequency for these QC samples are included in the applicable laboratory SOPs in Attachment 1. The acceptance criteria for all these QC checks are included the laboratory's SOPs. The acceptance criteria for MS/MSD samples and LCSs are also provided in Table 4.1 of this QAPP.

The laboratory QC requirements for phenolics include analyzing method blanks, initial and continuing calibration standards, LCSs, and MS/MSD samples. The laboratory QC

requirements for ammonia include analyzing method blanks, LCSs, and MS/MSD samples. The analysis frequency for these QC samples are included in the applicable laboratory SOPs in Attachment 1. The acceptance criteria these QC checks are included in the laboratory's SOPs. The acceptance criteria for MS/MSD samples and LCSs are also provided in Table 4.1 of this QAPP.

5.5 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS [2.2.6] B6

The procedures used to verify that instruments and equipment are functional and properly maintained are described in the following subsections.

5.5.1 FIELD INSTRUMENT MAINTENANCE [2.2.6] B6

There are no field instruments being used for the RA.

5.5.2 LABORATORY INSTRUMENT MAINTENANCE [2.2.6] B6

As part of their QA/QC program, the laboratory conducts a routine preventive maintenance program to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees will regularly perform routine scheduled maintenance and repair of (or coordinate with the instrument manufacturer for the repair of) all instruments. All maintenance that is performed will be documented in the laboratory's maintenance logbooks. All laboratory instruments are maintained in accordance with manufacturer's specifications.

Table 5.3 provides examples of the frequency at which components of key analytical instruments or equipment will be serviced. The SOPs in Attachment 1 provide complete details for instrument preventive maintenance.

5.6 CALIBRATION PROCEDURES AND FREQUENCY [2.2.7] B7

The procedures for maintaining the accuracy for all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses are described

in the following subsections. These instruments and equipment will be calibrated prior to each use or according to a periodic schedule.

5.6.1 FIELD INSTRUMENTS/EQUIPMENT [2.2.7] B7

There are no field instruments being used for the Phase 1 GW RA. Equipment to be used for field sampling will be examined to confirm that it is in operating condition.

5.6.2 LABORATORY INSTRUMENTS [2.2.7] B7

Calibration of laboratory equipment will be based on approved written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing quality control activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain a properly trained repair staff with in-house spare parts or will maintain service contracts with vendors.

The records of calibration will be kept as follows:

1. If possible, each instrument will have record of calibration permanently affixed with an assigned record number.
2. A logbook will be assigned to each instrument showing description, manufacturer, model numbers, date of last calibration and the signature of the person who calibrated the instrument, due date of next calibration and compensation or correction figures, as appropriate.
3. A written stepwise calibration procedure will be available for each piece of test and measurement equipment.
4. Any instrument that is not calibrated to the manufacturer's original specification will display a warning tag or will otherwise be removed from service, as appropriate.

Specific calibration procedures and frequencies are detailed in the laboratory SOPs in Attachment 1.

5.7 INSPECTION/ACCEPTANCE CRITERIA FOR SUPPLIES AND CONSUMABLES [2.2.8] B8

The procedures that will be used to ensure that supplies and consumables used in the field and laboratory will be available as needed and free of contaminants are detailed in the following subsections.

5.7.1 FIELD SUPPLIES AND CONSUMABLES [2.2.8] B8

Supplies and consumables for field sampling will be obtained from various vendors and include sample containers, preservatives, detergent and water for equipment decontamination, and rinsate blank water. The vendors and inspection and acceptance criteria for these field supplies were presented in Section 5.1.4 of this QAPP. Additional field supplies and consumables include pump tubing, and personnel protective equipment (PPE). Pump tubing will be constructed of pre-cleaned high density polyethylene. These materials will not introduce contaminants into the samples or interfere with the analyses. All field supplies will be consumed or replaced with sufficient frequency to prevent deterioration or degradation that may interfere with the analyses.

5.7.2 LABORATORY SUPPLIES AND CONSUMABLES [2.2.8] B8

EMT's vendor for general labware and reagents is Fisher Scientific. Vendors for metals and general chemistry parameters supplies and standards include Ultra Scientific and High Purity Standards. The lot numbers of reagents and standards are recorded and dates of receipt, first use, and expiration are documented. Certificates of analysis are maintained on file to document reagent/standard purity.

STAT's vendor for general labware is VWR. Vendors for reagents and standards are Ricca Chemicals, High Purity Standards, Inorganic Ventures, and CPI International. The lot numbers of reagents and standards are recorded and dates of receipt, first use, and expiration are documented. Certificates of analysis are maintained on file to document reagent/standard purity.

The SOPs in Attachment 1 provide details on identifying contaminants in reagents and standards, determining deterioration of reagents and standards, and the corrective actions required if contaminants or deterioration are identified. The laboratory QA Officer is ultimately responsible for the ensuring the acceptability of supplies and consumables.

5.8 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS) [2.2.9] B9

Historical data for the Site were generated during the various studies and monitoring events. Data generated during the RI and additional studies were validated by U.S. EPA Region 5 or its contractors.

5.9 DATA MANAGEMENT [2.2.10] B10

The procedures for managing data from generation to final use and storage are detailed in subsections that follow.

5.9.1 DATA RECORDING [2.2.10] B10

Field data will be recorded in field logbooks and will consist of sample collection conditions and sample identification. Field staff are responsible for recording field data and the Field QA Officer is responsible for identifying and correcting recording errors.

Laboratory data are recorded in a variety of formats. Data from instruments are recorded on magnetic media, strip charts, or bench sheets. The laboratory SOPs in Attachment 1 provide the data recording requirement for each preparation and analysis method.

5.9.2 DATA VALIDATION [2.2.10] B10

Validation of performance verification data for this project will primarily consist of checking for transcription errors and review of data recorded in field logbooks. Data transcribed from the field logbook into summary tables for reporting purposes will be

verified for correctness by the Field QA Officer or his designee. Any limitations on the use of performance verification data will be included in the RA reports.

Validation of the analytical data will be performed by BMcD's QA Officer or his designee based on the relevant and applicable evaluation criteria outlined in "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", EPA-540/R-04-004, October 2004. The evaluation and action criteria specified in this document (referred to hereafter as the National Functional Guidelines) will be used for validating the data. However, the acceptance limits for QC data will be the control limits determined statistically by the laboratory, not the control limits specified in the National Functional Guidelines. Qualifiers assigned to the data will be consistent with the data qualifiers specified in the National Functional Guidelines.

The following QC data deliverables will be evaluated on 100 percent of the data.

General Chemistry Analyses (ammonia and phenolics)

1. Technical Holding Times;
2. Blanks;
3. MS/MSD Results;
4. LCS Results;
5. Field Duplicates; and
6. Rinsate blank Samples.

Arsenic Analyses

1. Technical Holding Times;
2. Blanks;
3. MS/MSD results;
4. LCS Results;
5. Field Duplicates; and
6. Rinsate blank Samples.

The results of the data validation process will be documented in a memorandum that specifies all limitations on the usability of the analytical data.

5.9.3 DATA TRANSFORMATION/DATA REDUCTION [2.2.10] B10

Field data reduction procedures will be minimal in scope compared to those implemented for laboratory data. These data will be recorded into field logbooks immediately after the measurements are taken.

EMT will use the following protocol for data reduction procedures:

1. Raw data produced and checked by the responsible analyst.
2. The area supervisor or senior chemist reviews the data for attainment of quality control criteria established by the QAPP.
3. The area supervisor will decide whether any sample re-analysis is required.
4. Upon completion of all reviews and acceptance of the raw data by the area supervisor, a report will be generated and made available to the laboratory Project Manager.
5. The laboratory Project Manager will complete a thorough inspection of all reports.
6. Following review and approval of the preliminary report by the laboratory Project Manager, final reports will be generated and signed by the laboratory Director.

STAT will use the following protocol for data reduction procedures:

1. Raw data produced and checked by the responsible analyst and turned over for independent review by another analyst.
2. The area supervisor or senior chemist reviews the data for attainment of quality control criteria established by the QAPP.
3. The area supervisor will decide whether any sample re-analysis is required.
4. Upon completion of all reviews and acceptance of the raw data by the area supervisor, a report will be generated by one of the laboratory's Project Managers.

5. The laboratory Project Manager preparing the report will complete a thorough inspection of the report.
6. Following review and approval of the preliminary report by the laboratory Project Manager assigned to conduct review, final reports will be generated and signed by the laboratory Project Manager specifically designated as STAT Project Manager in this QAPP.

Specific equations used for data reduction are contained in each laboratory's SOPs in Attachment 1.

5.9.4 DATA TRANSMITTAL/TRANSFER [2.2.10] B10

Field data from sample collection will be recorded in the field logbook and may be entered into a standard Microsoft Excel spreadsheet format. BMcD's Field QA Officer is responsible for verifying the correctness of the field data that has been transferred to a spreadsheet format. Field data will be synchronized with geographical data from the Phase 1 GW RA design.

The laboratory will provide electronic data deliverables (EDDs) in the EQuIS 4-file format. EQuIS is an environmental data management software product by EarthSoft. BMcD uses Version 5 with Microsoft SQL Server as the database engine. The laboratory data are downloaded into the EDDs directly from the laboratory information management system (LIMS), thus eliminating the possibility of manual transcription errors. The EDDs are imported into EQuIS and the data are maintained in the database for manipulation and presentation.

Under the supervision of the BMcD QA Officer, a BMcD EQuIS database analyst will be responsible for verifying the correctness of the analytical database after the laboratory data for each sampling event have been imported. This is accomplished by comparing the data from the database to the hardcopy analytical report for a minimum of 10 percent of the sample results for each report. If discrepancies between the database and hardcopy analytical report are detected, a complete verification of the report will be performed or a new EDD will be submitted, imported, and verified as described previously.

5.9.5 DATA ANALYSIS [2.2.10] B10

Data will be analyzed as described in the Performance Standard Verification Plan, Attachment C to the Final Design.

5.9.6 DATA ASSESSMENT [2.2.10] B10

Assessment of laboratory data by the laboratory will be performed using the procedures detailed in the laboratories' SOPs in Attachment 1. These assessments included determining the mean, standard deviation, relative standard deviation, percent difference, RPD, and percent recovery for certain QC elements.

Assessment of QC data for data validation purposes will include determining the percent recovery, RPD, and percent completeness. The statistical equations to determine these parameters are provided in Section 7.3 of this QAPP.

5.9.7 DATA TRACKING [2.2.10] B10

Data generated in the field, such as sample identification, will be recorded in field logbooks. There are no unique or special tracking requirements for these data. The data will be transcribed for analysis and reporting and the original survey data and field logbooks will be maintained in the final evidence file.

Laboratory data tracking procedures are provided in the SOPs in Attachment 1. These SOPs provide the procedures for tracking data from generation to reporting, which is primarily conducted through each laboratory's LIMS. The laboratory Operations Manager is ultimately responsible for data tracking in the laboratory.

Tracking of analytical data in the EQuIS database includes recording the laboratory generating the data, the date when EDD was received and imported, the date when qualifiers were applied to the results, and the level of data validation performed. BMcD's Project Manager is ultimately responsible for tracking data from entry into the database to reporting.

5.9.8 DATA STORAGE AND RETRIEVAL [2.2.10] B10

EMT laboratory data will be stored by the laboratory in hardcopy format at their facility. Data are archived on site for a period of 5 years, after which time the data is disposed of via manifested document destruction subcontractors. Electronic instrument data are maintained on magnetic media (i.e., magnetic tape) for this same time period. The laboratory's Records Manager is responsible for data archiving and retrieval at their facility.

STAT laboratory data will be stored by the laboratory in hardcopy format at their facility. Data are archived on site for a period of 5 years,. Electronic instrument data are maintained on magnetic media (i.e., magnetic tape) for this same time period. The laboratory's Quality Assurance Director is responsible for hardcopy data archiving and retrieval at their facility. The laboratory's Technical Manager is responsible for electronic data archiving and retrieval at their facility.

BMcD's Project Manager is responsible for project data storage and retrieval. Field logbooks will be maintained in the site field office and each completed logbook will be transferred to the permanent project file in BMcD's Downers Grove, Illinois office for retention. At the conclusion of the Phase 1 GW RA, field logbooks associated with this task will be archived at BMcD's Downers Grove, Illinois office. Upon completion of the RA activities, the final evidence file will be archived at BMcD's Kansas City, Missouri headquarters.

All data including copies of the laboratory data will be retained for 10 years from the receipt of U.S. EPA's Certification of Completion of Remedial Action.

5.9.9 DATA SECURITY [2.2.10] B10

Laboratory data security is the responsibility of EMT's Records Manager and STAT's Quality Assurance Director. Archived data cannot be accessed without authorization. Each laboratory's LIMS is password protected and access rights are restricted by job function.

BMcD's data security procedures include limiting project database access to database analysts and general building security procedures including electronic key entry to work and file storage areas, and documentation of office visitors.

6.0 ASSESSMENT/OVERSIGHT [2.3]

The following subsections describe the procedures used to ensure proper implementation of this QAPP and the activities for assessing the effectiveness of the implementation of the project and associated QA/QC activities.

6.1 ASSESSMENTS AND RESPONSE ACTIONS [2.3.1] C1

Assessments consisting of internal and external audits will be performed during the project. Internal technical system audits of both field and laboratory procedures will be conducted to verify that sampling and analysis are being performed in accordance with the procedures established in the FSP and QAPP. External field and laboratory audits may be conducted by U.S. EPA.

An internal field technical system audit of field activities will be conducted by the Field QA Officer or his designee at the beginning of the field sampling activities to identify deficiencies in the field sampling and documentation procedures. The field technical system audit will include examining field sampling records, and chain-of-custody documentation. In addition, sample collection, handling, and packaging in compliance with the established procedures will be reviewed during the field audit. Any deficiencies identified will be documented and corrective actions will be taken to rectify the deficiencies.

Corrective action resulting from internal field technical system audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The Field QA Officer will identify deficiencies and recommended corrective action to the Project Manager. Implementation of corrective actions will be performed by the Field QA Officer and field team. Corrective action will be documented in the field logbook and/or the project file. Follow-up audits will be performed as necessary to verify that deficiencies have been corrected, and that the QA/QC procedures described in this QAPP and the FSP are maintained throughout the project.

An external field technical system audit may be conducted by U.S. EPA Region 5 FSS any time during the field operations. These audits may or may not be announced and are conducted at the discretion of U.S. EPA Region 5.

An internal laboratory technical system audit will be conducted by the laboratory QA Officer or her designee. The laboratory technical system audit is conducted on an annual basis and includes examining laboratory documentation regarding sample receiving, sample log-in, storage and tracking, chain-of-custody procedures, sample preparation and analysis, instrument operating records, data handling and management, data tracking and control, and data reduction and verification. The laboratory QA Officer will evaluate the results of the audit and provide a final report to section managers and the laboratory Director (EMT) or laboratory President (STAT) that includes any deficiencies and/or noteworthy observations.

Corrective action resulting from deficiencies identified during the internal laboratory technical system audit will be implemented immediately. The laboratory Director (EMT) or laboratory President (STAT), in consultation with the section leader, laboratory supervisor and staff, will approve the required corrective action to be implemented by the laboratory staff. The laboratory QA/QC Officer will ensure implementation and documentation of the corrective action. All problems requiring corrective action and the corrective action taken will be reported to the laboratory Director (EMT) or laboratory President (STAT). Follow-up audits will be performed as necessary to verify that deficiencies have been corrected, and that the QA/QC procedures described in the QAPP are maintained throughout the project.

An external laboratory audit may be conducted by U.S. EPA Region 5 FSS personnel. These audits may or may not be announced and are at the discretion of U.S. EPA Region 5. The external laboratory audits will include, but not be limited to, reviewing laboratory analytical procedures, laboratory on-site audits, and/or submitting performance evaluation samples to the laboratory for analysis.

An external laboratory audit may be conducted at least once prior to the initiation of the sampling and analysis activities.

6.2 REPORTS TO MANAGEMENT [2.3.2] C2

Quality Assurance Management Reports will be prepared during the RA activities. These QA Management Reports will be included with the monthly progress reports that are submitted to U.S. EPA and IEPA when data gathering or assessment activities are being conducted. Minimally, these reports will include project status, results of performance evaluations and system audits, results of periodic data quality validation and assessment and data use limitations, and any significant QA problems identified and corrective actions taken.

BMcD's QA Officer will be responsible within the organizational structure for preparing these reports. BMcD's Project Manager will be provided with these reports for distribution with monthly status reports. The pre-design study report will also include a separate QA/QC section that will summarize data quality information contained in the periodic QA Management Reports and provides an overall data quality assessment compared to the data quality objectives outlined in this QAPP.

7.0 DATA VERIFICATION/VALIDATION AND USABILITY [2.4]

The QA activities that will be performed to ensure that the RA data are scientifically defensible, properly documented, of known quality, and meet the project objectives are described in the following sections.

7.1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS [2.4.1] D1

All field and laboratory data for performance verification will be reviewed and verified/validated. The procedures and criteria used to verify and validate field and laboratory data will consist of evaluating the data to the measurement performance criteria in Section 4.2 of this QAPP. Field data and logbooks will be reviewed to ensure that the requirements of the sampling program, including the number of samples and locations, sampling procedures, and sample handling, were fulfilled. Acceptable departures from the planned sampling program will not impact the data usability.

Sample collection procedures will be reviewed for compliance with the requirements of the FSP and QAPP. If alternate sampling procedure were used, the acceptability of the procedure will be evaluated to determine the affect on the usability of the data. Data usability will not be affected if the procedure used is determined to be an acceptable alternative that fulfills the measurement performance criteria in Section 4.2 of this QAPP. However, data generated from sampling procedures that do not provide representative samples will be rejected.

Sample handling records will be reviewed to ensure that sample integrity remained intact from collection to laboratory receipt and that samples were properly preserved. Chain-of-custody documentation and sample condition upon laboratory receipt will be reviewed. The data from samples for which the chain-of-custody or sample identification cannot be verified will be rejected. The data for samples that were not properly preserved will be qualified or rejected depending on the severity of the deviation from the requirements of the FSP and QAPP. The criteria for rejecting improperly preserved samples will be that the sample has been rendered unsuitable for analysis. The criteria for qualifying or rejecting data for samples that are received at the laboratory without being properly preserved, but not rendered unsuitable for analysis, will be based on the sample

holding time period evaluation criteria for unpreserved samples specified in the National Functional Guidelines. Data qualification will be consistent with the action specified in the National Functional Guidelines.

Field and laboratory data will be verified to ensure that the methods used to analyze the samples were consistent with the requirements of this QAPP. Data generated from the use of unapproved methods will be rejected.

QC data will be reviewed to determine compliance with the acceptance criteria in Section 4.2 of this QAPP. QC data that do not meet the acceptance criteria will result in sample data qualification. Significant departures from the QC acceptance criteria may result in rejected data. Situations that result in data rejection include samples analyzed beyond twice the technical holding time period, inorganic LCS analyte recoveries less than 50 percent if the analyte is not detected in the associated samples, and inorganic matrix spike analyte recoveries less than 30 percent if the analyte is not detected in the associated samples.

7.2 VERIFICATION AND VALIDATION METHODS [2.4.2] D2

Field data will be verified by reviewing field documentation and chain-of-custody records. The laboratory will internally verify the laboratory data by reviewing and documenting sample receipt, sample preparation, sample analysis (including internal QC checks), data reduction and reporting. Any deviations from the acceptance criteria, corrective actions taken, and data determined to be of limited usability (i.e., laboratory-qualified data) will be noted in the case narrative of the laboratory report.

Data validation will be conducted by BMcD consistent with the procedure identified in Section 5.9.2 of this QAPP. The data verification/validation procedure will identify data as being acceptable, of limited usability (qualified as estimated), or rejected. The conditions that result in data being qualified as estimated or rejected are identified in Section 7.1 of this QAPP. The results of the data verification/validation will be provided in data validation memoranda that are provided to BMcD's Project Manager and are included in Quality Assurance Management Reports.

Data determined to be unusable may require corrective action to be taken. Potential types of corrective action may include re-sampling by the field team or reanalysis of samples by the laboratory. The corrective actions taken are dependent upon the ability to mobilize the field team and whether the data are critical for project DQOs to be achieved. Should BMcD's QA Officer identify a situation requiring corrective action during data verification/validation, BMcD's Project Manager will be responsible for approving the implementation of the corrective action.

7.3 USABILITY/RECONCILIATION WITH DATA QUALITY OBJECTIVES [2.4.3] D3

The overall usability of the data for the RA activities will be assessed by evaluating the PARCCS of the data set to the measurement performance criteria in Section 4.2 of this QAPP using basic statistical quantities as applicable. The procedures and statistical formulas to be used for these evaluations are presented in the following subsections.

7.3.1 PRECISION [2.4.3] D3

Project precision will be evaluated by assessing the RPD data from field duplicate samples. Analytical precision will be evaluated by assessing the RPD data from either duplicate spiked sample analyses or duplicate sample analyses. The RPD between two measurements is calculated using the following simplified formula:

$$RPD\ (\%) = \frac{|\ R_1 - R_2\ |}{(R_1 + R_2)/2} \times 100$$

where:

R_1 = value of first result
R_2 = value of second result

Overall precision for the sampling programs will be determined by calculating the mean RPD for all field duplicates in a given sampling program. This will provide an evaluation of the overall variability attributable to the sampling procedure, sample matrix, and laboratory procedures in each sampling program.

The overall precision requirement will be the same as the project precision. It should be noted that the RPD of two measurements can be very high when the data approach the quantitation limit of an analysis. The calculation of the mean RPD will only include the RPD values for field duplicate sample analyte data that are greater than or equal to 5 times the quantitation limit for an analysis. An RPD of 35 percent for groundwater sample field duplicates will be used as an advisory limit.

7.3.2 ACCURACY/BIAS [2.4.3] D3

The data from method/preparation blank samples, rinsate blank samples, MS/MSD samples, and LCSs will be used to determine accuracy and potential bias of the sample data.

The data from method/preparation blank samples provide an indication of laboratory contamination that may result in bias of sample data. Sample data associated with method/preparation blank contamination will have been identified during the data verification/validation process. Sample data associated with method/preparation blank contamination are evaluated during data validation procedure to determine if analytes detected in the samples and the associated method/preparation blanks are "real" or are the result of laboratory contamination. The procedure for this evaluation involves comparing the concentration of the analyte in the sample to the concentration in the method/preparation blank taking into account adjustments for sample dilutions and dry-weight reporting. In general, the sample data are qualified as not detected if the sample concentration is less than 5 times (10 times for common laboratory contaminants) the method/preparation blank concentration. Typically, the quantitation limit for the affected analyte is elevated to the concentration detected in the sample.

The data from rinsate blanks provide an indication of field conditions that may result in bias of sample data. Sample data associated with contaminated rinsate blank samples will have been identified during the data verification/validation process. The evaluation procedure and qualification of sample data associated with rinsate blank contamination is performed in the same manner as the evaluation procedure for method blank sample contamination.

Matrix spike sample data provide information regarding the accuracy/bias of the analytical methods relative to the sample matrix. Matrix spike samples are field samples that have been fortified with target analytes prior to sample preparation and analysis. The percent recovery data provide an indication of the effect that the sample matrix may have on the preparation and analysis procedure. Sample data exhibiting matrix effects will have been identified during the data verification/validation process.

Analytical accuracy/bias will be determined by evaluating the percent recovery data of LCSs. LCSs are artificial samples prepared in the laboratory using a blank matrix that is fortified with analytes from a standard reference material that is independent of the calibration standards. LCSs are prepared and analyzed in the same manner as the field samples. The data from LCS analyses will provide an indication of the accuracy and bias of the analytical method for each target analyte.

Percent recovery (%R) is calculated using the following formula:

$$\%R = \frac{SSR - SR}{SA} \quad X \ 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

The percent recovery of LCSs samples are determined by dividing the measured value by the true value and multiplying by 100.

Overall accuracy/bias for the sampling events will be determined by calculating the percent of accuracy measurements that meet the measurement performance criteria specified in Section 4.2 of this QAPP. Overall accuracy will be considered acceptable if the MS/MSD percent recoveries and LCS percent recoveries are met for at least 75 percent of the samples.

7.3.3 SAMPLE REPRESENTATIVENESS [2.4.3] D3

Representativeness of the samples will be assessed by reviewing the results of field audits and the data from field duplicate samples. Overall sample representativeness will be determined by calculating the percent of field duplicate sample data that achieved the RPD criteria specified in Section 4.2 of this QAPP. Overall sample representativeness will be considered acceptable if the results of field audits indicate that the approve sampling methods or alternate acceptable sampling methods were used to collect the samples and the field duplicate RPD data are acceptable for at least 75 percent of the samples.

7.3.4 COMPLETENESS [2.4.3] D3

Completeness will be assessed by comparing the number of valid (usable) sample results to the total possible number of results within a specific sample matrix and/or analysis. Percent completeness will be calculated using the following formula:

$$\% \text{ Completeness} = \frac{\text{Number of Valid (usable) measurements}}{\text{Number of Measurements Planned}} \times 100$$

Overall completeness will be assessed by calculating the mean percent completeness for the entire set of data obtained for each sampling program. The overall completeness for the RA will be calculated when all sampling and analysis is concluded. Overall completeness will be considered acceptable if at least 90 percent of the data are determined to be valid.

7.3.5 COMPARABILITY [2.4.3] D3

The comparability of data sets will be evaluated by reviewing the sampling and analysis methods used to generate the data for each data set. Project comparability will be determined to be acceptable if the sampling and analysis methods specified in this QAPP and any approved QAPP revisions or amendments are used for generating the data.

Overall comparability of data from split samples (samples that are collected at the same time from the same location and split equally between two parties using sample

containers from the same source or vendor) will be evaluated by determining the RPD of detected analytes in both samples following data verification/validation. Analytes that are detected in only one of the two samples will be assessed by reviewing the data verification/validation reports for both data sets and determining the cause of the discrepancy. Overall comparability of split sample data will be considered acceptable if the RPD for detected analytes with concentrations greater than or equal to 5 times their respective quantitation limits does not exceed RPD acceptance criteria for field duplicate samples (i.e., 35 percent for groundwater sample duplicates).

7.3.6 SENSITIVITY AND QUANTITATION LIMITS [2.4.3] D3

The quantitation limits for the sample data will be reviewed to ensure that the sensitivity of the analyses was sufficient to achieve the groundwater clean-up criteria. The method/preparation blank sample data and LCSs percent recovery data will be reviewed to assess compliance with the measurement performance criteria specified in Section 4.2 of this QAPP.

Overall sensitivity will be assessed by comparing the sensitivity to the detectability requirements for the analyses. Overall sensitivity will be considered acceptable if quantitation limits for the samples are less than the applicable evaluation criteria.

It should be noted that quantitation limits may be elevated as a result of high concentrations of target compounds, non-target compounds, and matrix interferences (collectively known as sample matrix effects). In these cases, the sensitivity of the analyses will be evaluated on an individual sample basis relative to the applicable evaluation criteria. The need to investigate the use of alternate analytical methods may be required if the sensitivity of the analytical methods identified in this QAPP cannot achieve the evaluation criteria as a result of sample matrix effects.

7.3.7 DATA LIMITATIONS AND ACTIONS [2.4.3] D3

Data use limitations will be identified in data validation memoranda or Quality Assurance Management reports. Data that do not meet the measurement performance criteria specified in this QAPP will be identified and the impact on the project quality objectives

will be assessed and discussed in these reports. Specific actions for data that do not meet the measurement performance criteria depends on the use of the data, and may require that additional samples are collected or the use of the data be restricted. Any alternative methods of collection or analysis must be approved by the regional project manager from the U.S. EPA Region 5 prior to implementation.

Determination of the overall data quality for a specific sampling program will be conducted at the completion of the program. Data validation memoranda or Quality Assurance Management reports will be included with the project reports identified in the Final Design Report.

Figure 2.1
Project Team Organization Chart
Remedial Action/Groundwater Treatment
Waukegan Manufactured Gas and Coke Plant Site

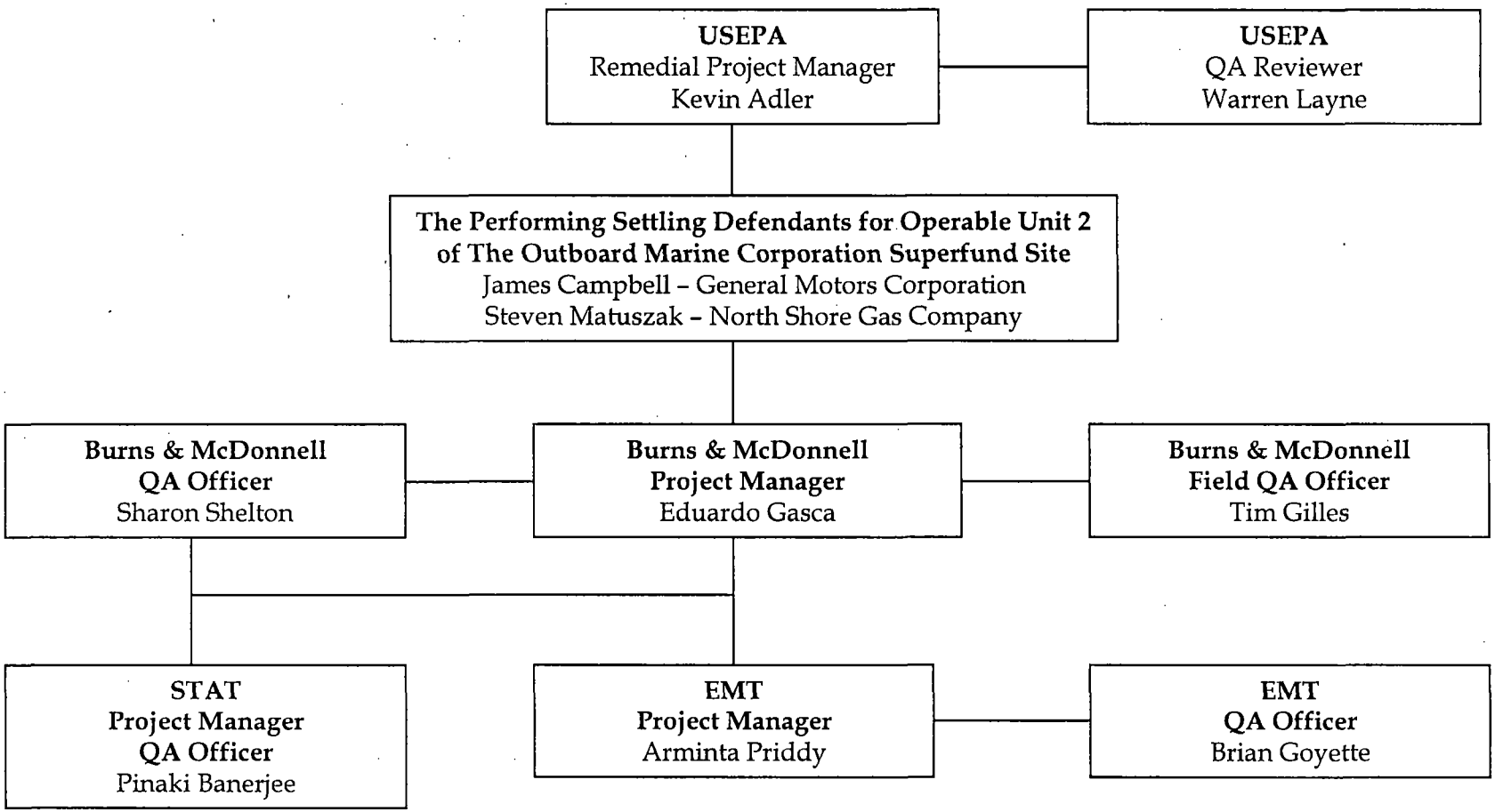


Fig. 5.1A - EMT Sample Container Certificate of Analysis Example

QEC
Quality Environmental Containers

C-QEC
CERTIFICATE OF QUALITY ENVIRONMENTAL COMPLIANCE

P.O. Box 1160 • Beaver, WV 25813 • 800-255-3950 • 304-255-3900

Lot Number **A-8-294-01DB** **32 oz. Amber Packer 2122-0032**

The above lot number has been specially cleaned using procedures specified by the USEPA to *limit the concentration of the following organic compounds:*

Compound	CRQL (µg/L)	Compound	CRQL (µg/L)	Compound	CRQL (µg/L)
Chloromethane	1.0	Ethylbenzene	1.0	Acenaphthylene	5.0
Bromomethane	1.0	Styrene	1.0	2,6-Dinitrotoluene	5.0
Vinyl Chloride	1.0	Xylenes(total)	1.0	3-Nitroaniline	20.0
Chloroethane	1.0	1,3-Dichlorobenzene	1.0	Acenaphthene	5.0
Methylene Chloride	2.0	1,4-Dichlorobenzene	1.0	2,4-Dinitrophenol	20.0
Acetone	5.0	1,2-Dichlorobenzene	1.0	4-Nitrophenol	20.0
Carbon Disulfide	1.0	1,2-Dibromo-3-chloropropane	1.0	Dibenzofuran	5.0
1,1-Dichloroethene	1.0	Phenol	5.0	2,4-Dinitrotoluene	5.0
1,1-Dichloroethane	1.0	bis-(2-Chloroethyl)ether	5.0	Diethylphthalate	5.0
cis-1,2-Dichloroethene	1.0	2-Chlorophenol	5.0	4-Chlorophenyl-phenylether	5.0
trans-1,2-Dichloroethene	1.0	2-Methylphenol	5.0	Fluorene	5.0
Chloroform	1.0	2,2'-oxybis-(1-Chloropropane)	5.0	4-Nitroaniline	20.0
1,2-Dichloroethane	1.0	4-Methylphenol	5.0	4,6-Dinitro-2-methylphenol	20.0
2-Butanone	5.0	N-Nitroso-di-n-dipropylamine	5.0	N-Nitrosodiphenylamine	5.0
Bromochloromethane	1.0	Hexachloroethane	5.0	4-Bromophenyl-phenylether	5.0
1,1,1-Trichloroethane	1.0	Nitrobenzene	5.0	Hexachlorobenzene	5.0
Carbon Tetrachloride	1.0	Isophorone	5.0	Pentachlorophenol	20.0
Bromodichloromethane	1.0	2-Nitrophenol	5.0	Phenanthrene	5.0
1,2-Dichloropropane	1.0	2,4-Dimethylphenol	5.0	Anthracene	5.0
cis-1,3-Dichloropropene	1.0	bis-(2-Chloroethoxy)methane	5.0	Di-n-butylphthalate	5.0
Trichloroethene	1.0	2,4-Dichlorophenol	5.0	Fluoranthene	5.0
Dibromochloromethane	1.0	1,2,4-Trichlorobenzene	5.0	Pyrene	5.0
1,1,2-Trichloroethane	1.0	Naphthalene	5.0	Butylbenzylphthalate	5.0
Benzene	1.0	4-Chloroaniline	5.0	3,3'-Dichlorobenzidine	5.0
trans-1,3-Dichloropropene	1.0	Hexachlorobutadiene	5.0	Benz[a]anthracene	5.0
Bromoform	1.0	4-Chloro-3-methylphenol	5.0	Chrysene	5.0
4-Methyl-2-pentanone	5.0	2-Methylnaphthalene	5.0	bis-(2-Ethylhexyl)phthalate	5.0
2-Hexanone	5.0	Hexachlorocyclopentadiene	5.0	Di-n-octylphthalate	5.0
Tetrachloroethene	1.0	2,4,6-Trichlorophenol	5.0	Benzo[b]fluoranthene	5.0
1,1,2,2-Tetrachloroethane	1.0	2,4,5-Trichlorophenol	20.0	Benzo[k]fluoranthene	5.0
1,2-Dibromoethane	1.0	2-Chloronaphthalene	5.0	Benzo[a]pyrene	5.0
Toluene	1.0	2-Nitroaniline	20.0	Indeno(1,2,3-cd)pyrene	5.0
Chlorobenzene	1.0	*Dimethylphthalate*	5.0	Dibenz[a,h]anthracene	5.0
				Benzo[g,h,i]perylene	5.0

The above lot number has also been specially cleaned using procedures specified by the USEPA to limit the concentration of the following pesticides/PCBs compounds:

Compound	CRQL (µg/L)	Compound	CRQL (µg/L)
alpha-BHC	0.01	4,4'-DDT	0.02
beta-BHC	0.01	Methoxychlor	0.10
delta-BHC	0.01	Endrin ketone	0.02
gamma-BHC (Lindane)	0.01	Endrin aldehyde	0.02
Heptachlor	0.01	alpha-Chlordane	0.01
Aldrin	0.01	gamma-chlordane	0.01
Heptachlor epoxide	0.01	Toxaphene	1.0
Endosulfan I	0.01	Aroclor-1016	0.20
Dieldrin	0.02	Aroclor-1221	0.20
4,4'-DDE	0.02	Aroclor-1232	0.40
Endrin	0.02	Aroclor-1242	0.20
Endosulfan II	0.02	Aroclor-1248	0.20
4,4'-DDD	0.02	Aroclor-1254	0.20
Endosulfan sulfate	0.02	Aroclor-1260	0.20

The above lot number has also been specially cleaned using procedures specified by the USEPA to limit the concentration of the following elements:

Element	CRQL (µg/L)	Element	CRQL (µg/L)
Aluminum	100	Manganese	10
Antimony	5	Mercury	0.2
Arsenic	2	Nickel	20
Barium	20	Potassium	750
Beryllium	1	Selenium	3
Cadmium	1	Silver	10
Calcium	500	*Sodium*	*500*
Chromium	10	Thallium	10
Cobalt	10	Vanadium	10
Copper	10	Zinc	20
Iron	500	Cyanide	10
Lead	2	*Fluoride*	*200*
Magnesium	500	Nitrate/Nitrite	100
		Oil & Grease	1000

Fig. 5.1B - STAT Sample Container Certificate of Analysis Example

C-QEC

CERTIFICATE OF QUALITY ENVIRONMENTAL COMPLIANCE

Quality Environmental Containers P.O. Box 1160 • Beaver, WV 25813 • 800-255-3950 • 304-255-3900

Lot Number **H-8-274-01DB** **1000 ml Amber Boston Round 2121-0033**

The above lot number has been specially cleaned using procedures specified by the USEPA to limit the concentration of the following organic compounds:

Compound	CRQL (µg/L)	Compound	CRQL (µg/L)	Compound	CRQL (µg/L)
Chloromethane	1.0	Ethylbenzene	1.0	Acenaphthylene	5.0
Bromomethane	1.0	Styrene	1.0	2,6-Dinitrotoluene	5.0
Vinyl Chloride	1.0	Xylenes(total)	1.0	3-Nitroaniline	20.0
Chloroethane	1.0	1,3-Dichlorobenzene	1.0	Acenaphthene	5.0
Methylene Chloride	2.0	1,4-Dichlorobenzene	1.0	2,4-Dinitrophenol	20.0
Acetone	5.0	1,2-Dichlorobenzene	1.0	4-Nitrophenol	20.0
Carbon Disulfide	1.0	1,2-Dibromo-3-chloropropane	1.0	Dibenzofuran	5.0
1,1-Dichloroethene	1.0	Phenol	5.0	2,4-Dinitrotoluene	5.0
1,1-Dichloroethane	1.0	bis-(2-Chloroethyl)ether	5.0	Diethylphthalate	5.0
cis-1,2-Dichloroethene	1.0	2-Chlorophenol	5.0	4-Chlorophenyl-phenylether	5.0
trans-1,2-Dichloroethene	1.0	2-Methylphenol	5.0	Fluorene	5.0
Chloroform	1.0	2,2'-oxybis-(1-Chloropropane)	5.0	4-Nitroaniline	20.0
1,2-Dichloroethane	1.0	4-Methylphenol	5.0	4,6-Dinitro-2-methylphenol	20.0
2-Butanone	5.0	N-Nitroso-di-n-dipropylamine	5.0	N-Nitrosodiphenylamine	5.0
Bromochloromethane	1.0	Hexachloroethane	5.0	4-Bromophenyl-phenylether	5.0
1,1,1-Trichloroethane	1.0	Nitrobenzene	5.0	Hexachlorobenzene	5.0
Carbon Tetrachloride	1.0	Isophorone	5.0	Pentachlorophenol	20.0
Bromodichloromethane	1.0	2-Nitrophenol	5.0	Phenanthrene	5.0
1,2-Dichloropropane	1.0	2,4-Dimethylphenol	5.0	Anthracene	5.0
cis-1,3-Dichloropropene	1.0	bis-(2-Chloroethoxy)methane	5.0	Di-n-butylphthalate	5.0
Trichloroethene	1.0	2,4-Dichlorophenol	5.0	Fluoranthene	5.0
Dibromochloromethane	1.0	1,2,4-Trichlorobenzene	5.0	Pyrene	5.0
1,1,2-Trichloroethane	1.0	Naphthalene	5.0	Butylbenzylphthalate	5.0
Benzene	1.0	4-Chloroaniline	5.0	3,3'-Dichlorobenzidine	5.0
trans-1,3-Dichloropropene	1.0	Hexachlorobutadiene	5.0	Benz[a]anthracene	5.0
Bromoform	1.0	4-Chloro-3-methylphenol	5.0	Chrysene	5.0
4-Methyl-2-pentanone	5.0	2-Methylnaphthalene	5.0	bis-(2-Ethylhexyl)phthalate	5.0
2-Hexanone	5.0	Hexachlorocyclopentadiene	5.0	Di-n-octylphthalate	5.0
Tetrachloroethene	1.0	2,4,6-Trichlorophenol	5.0	Benzo[b]fluoranthene	5.0
1,1,2,2-Tetrachloroethane	1.0	2,4,5-Trichlorophenol	20.0	Benzo[k]fluoranthene	5.0
1,2-Dibromoethane	1.0	2-Chloronaphthalene	5.0	Benzo[a]pyrene	5.0
Toluene	1.0	2-Nitroaniline	20.0	Indeno(1,2,3-cd)pyrene	5.0
Chlorobenzene	1.0	Dimethylphthalate	5.0	Dibenz[a,h]anthracene	5.0
				Benzo[g,h,i]perylene	5.0

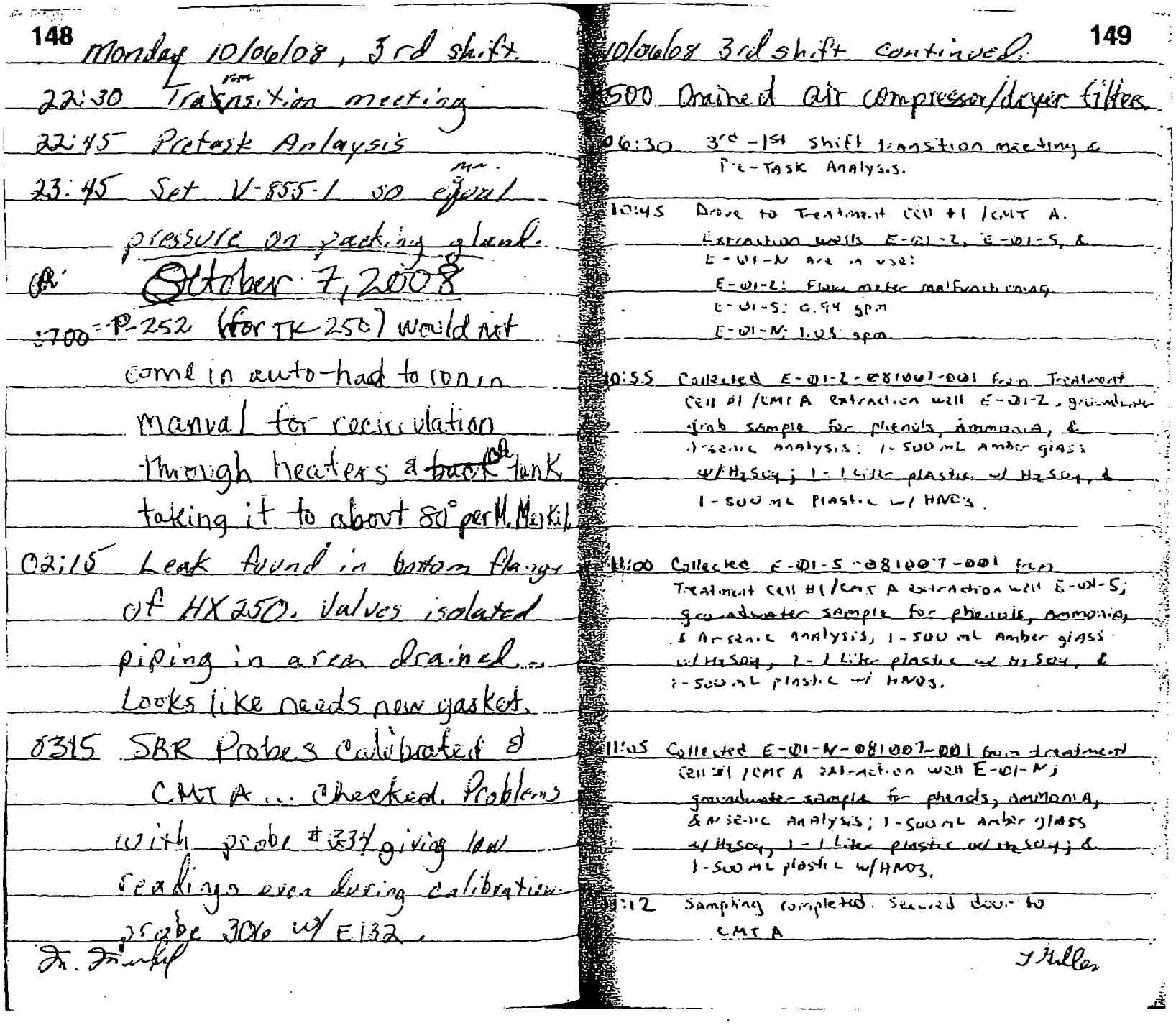
The above lot number has also been specially cleaned using procedures specified by the USEPA to limit the concentration of the following pesticides/PCBs compounds:

Compound	CRQL (µg/L)	Compound	CRQL (µg/L)
alpha-BHC	0.01	4,4'-DDT	0.02
beta-BHC	0.01	Methoxychlor	0.10
delta-BHC	0.01	Endrin Ketone	0.02
gamma-BHC (Lindane)	0.01	Endrin aldehyde	0.02
Heptachlor	0.01	alpha-Chlordane	0.01
Aldrin	0.01	gamma-chlordane	0.01
Heptachlor epoxide	0.01	Toxaphene	1.0
Endosulfan I	0.01	Aroclor-1016	0.20
Dieldrin	0.02	Aroclor-1221	0.20
4,4'-DDE	0.02	Aroclor-1232	0.40
Endrin	0.02	Aroclor-1242	0.20
Endosulfan II	0.02	Aroclor-1248	0.20
4,4'-DDD	0.02	Aroclor-1254	0.20
Endosulfan sulfate	0.02	Aroclor-1260	0.20

The above lot number has also been specially cleaned using procedures specified by the USEPA to limit the concentration of the following elements:

Element	CRQL (µg/L)	Element	CRQL (µg/L)
Aluminum	100	Manganese	10
Antimony	5	Mercury	0.2
Arsenic	2	Nickel	20
Barium	20	Potassium	750
Beryllium	1	Selenium	3
Cadmium	1	Silver	10
Calcium	500	Sodium	500
Chromium	10	Thallium	10
Cobalt	10	Vanadium	10
Copper	10	Zinc	20
Iron	500	Cyanide	10
Lead	2	Fluoride	200
Magnesium	500	Nitrate/Nitrite	100
		Oil & Grease	1000

Figure 5.2 - Field Log Book Example



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Monday 10/06/08, 3rd shift.
22:30 Transition meeting
22:45 Pretask Anlaysis
23:45 Set V-855-1 so equal pressure on packing gland.
Oct 7 October 7, 2008
0700 P-252 (for TK-250) would not come in auto-had to run in manual for recirculation through heaters & back to tank taking it to about 80° per H. Merkl.
02:15 Leak found in bottom flange of HX250. Valves isolated piping in area drained... Looks like needs new gasket.
0315 SBR Probes calibrated & CMT A... checked. Problems with probe #334 giving bad readings even during calibration. probe 306 w/ E132.
T. Tuttle

10/06/08 3rd shift continued. 149

0500 Drained air compressor/dryer filters

06:30 3rd-1st shift transition meeting & Pre-Task Analysis.

10:45 Drove to Treatment Cell #1 / CMT A. Extraction wells E-01-2, E-01-5, & E-01-N are in use:
E-01-2: Flow meter malfunctioning
E-01-5: 6.94 gpm
E-01-N: 1.03 gpm

10:55 Collected E-01-2-081007-001 from Treatment Cell #1 / CMT A extraction well E-01-2, groundwater grab sample for phenols, ammonia, & Arsenic analysis; 1-500 mL Amber glass w/ H_2SO_4; 1-1 Liter plastic w/ H_2SO_4, & 1-500 mL Plastic w/ HNO_3.

11:00 Collected E-01-5-081007-001 from Treatment Cell #1 / CMT A extraction well E-01-5; groundwater sample for phenols, ammonia, & Arsenic analysis, 1-500 mL Amber glass w/ H_2SO_4, 1-1 Liter plastic w/ H_2SO_4, & 1-500 mL plastic w/ HNO_3.

11:05 Collected E-01-N-081007-001 from treatment Cell #1 / CMT A extraction well E-01-N; groundwater sample for phenols, ammonia, & Arsenic Analysis; 1-500 mL Amber glass w/ H_2SO_4, 1-1 Liter plastic w/ H_2SO_4; & 1-500 mL plastic w/ HNO_3.

11:12 Sampling completed. Secured door to CMT A

T Miller

TABLE 3.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

Location	*Sample Matrix*	*Laboratory Parameters*	*Number of Samples*	*QC Samples*			*Total*
				Field Blanks	*Field Duplicates*	*MS/MSD*[1]	
Groundwater	Water	Arsenic, Ammonia, Phenolics					
Normal Cell Operation			350	18	18	18	404
Cell 4			230	12	12	12	266
Injection			260	13	13	13	299

Note:

1. Matrix spike/matrix duplicate (MS/MSD) analyses will be performed for all analytes. MS/MSD samples will be collected at a frequency of 1 per 20 or fewer remedial action samples.

TABLE 3.2

TARGETED QUANTITATION LIMITS
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

Water Sample Analyses	*Targeted Quantitation Limit (mg/L)*	*Cleanup Objective (mg/L)*
General Chemistry		
Ammonia	0.1	<3
Phenolics (4-AAP)	0.1	<1
Total Metals		
Arsenic	0.01	<1

TABLE 4.1

PERCENT RECOVERY AND RELATIVE PERCENT DIFFERENCE CONTROL LIMITS
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

ENVIRONMENTAL MONITORING AND TECHNOLOGIES, INC.

Water Sample Analyses	*Control Limits*		
	MS/MSD		*LCS*
	% Recovery	*RPD*	*% Recovery*
General Chemistry			
Ammonia	85-115	10	90-110
Phenolics (4-AAP)	80-115	10	90-110
Total Metals			
Arsenic (200.7)	83.3-106	5.43	81.3-118
Arsenic (200.8)	70-130	20	91.3-114

STAT ANALYSIS CORPORATION

Water Sample Analyses	*Control Limits*		
	MS/MSD		*LCS*
	% Recovery	*RPD*	*% Recovery*
General Chemistry			
Ammonia	75-125	20	80-120
Phenolics (4-AAP)	75-125	20	80-120
Total Metals			
Arsenic	75-125	20	80-120

TABLE 5.1

CONTAINER, PRESERVATION, SHIPPING, AND PACKAGING REQUIREMENTS
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

Water Analyses	*Sample Containers*[1]	*Preservation*[2]	*Maximum Holding Time from Sample Collection*[3]	*Volume of Sample*	*Shipping*	*Normal Packaging*
Total Arsenic	One 250 mL polyethylene bottle	HNO_3 to pH< 2	6 months for analysis	Fill to neck of bottle	Laboratory Courier	Iced Cooler
Ammonia	One 250 mL polyethylene or glass bottle	H2SO4 to pH< 2	28 days to analysis	Fill to neck of bottle	Laboratory Courier	Iced Cooler
Phenolics (4-AAP)	One 250 mL or pint glass bottle	H2SO4 to pH< 2	28 days to analysis	Fill to neck of bottle	Laboratory Courier	Iced Cooler

Notes:
1. To the extent possible, analyses will be combined to minimize the sample containers required. One 1-L or 1-Qt. glass bottle preserved with H_2So_4 to pH<2 can be used for both ammonia and phenolics.
2. Samples requiring refrigeration will be shipped with bagged, cubed ice, and will be stored at 4°± 2°C following laboratory receipt and log-in.
3. The holding times presented are technical holding times that are based on the time from sample collection.

TABLE 5.2

SUMMARY OF ANALYTICAL METHODS
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

ENVIRONMENTAL MONITORING AND TECHNOLOGIES, INC.

Water Analyses

Parameter	*Preparation Method*	*Analysis Method*
Total Arsenic	SOP #101 for SW-846 3015B	SOP #111 for EPA(a) 200.7 SOP #118 for EPA(a) 200.8
Ammonia*	SOP #004 for SM 4500 NH_3 E	SOP #004 for SM 4500 NH_3 E
Phenolics (4-AAP)	SOP #036 for SW-846 9065/EPA(b) 420.1	SOP #036 for SW-846 9065/EPA(b) 420.1

STAT ANALYSIS CORPORATION

Water Analyses

Parameter	*Preparation Method*	*Analysis Method*
Total Arsenic	SOP #3005 for SW-846 3005A	SOP #4510 for SW-846 6020
Ammonia*	SOP #3250 for SM 4500 NH_3 B	SOP #4250 for SM 4500 NH_3 H and C
Phenolics (4-AAP)	SOP #3620 for SW-846 9065	SOP #4715 for SW-846 9066

* EMT references Method 4500 NH_3 E - Titrimetric Method from the 18th Edition of *Standard Methods* (SM) in their SOP. Ammonia methods have been renumbered in subsequent editions. STAT references the same titrimetric method (4500 NH_3 C) from the 20th Edition.

References:

EPA(a) - Methods for the Determination of Metals in Environmental Samples, June 1991, EPA Publication No. EPA-600/4-91-010.
EPA(b) - "Methods for Chemical Analysis of Water and Wastes", March 1983. EPA Publication No. 600/4-79-020.
SW-846 - "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods", Third Ed., September 1986, as amended by Updates I through IVB EPA Publication No. SW-846.
SM - *Standard Methods for the Examination of Water and Wastewater,* various Eds. Jointly prepared and published by the American Public Health Association, American Water Works Association, and Water Environment Federation.

TABLE 5.3A

ROUTINE PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

ENVIRONMENTAL MONITORING AND TECHNOLOGIES, INC.

Instrument	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Inductively Coupled Plasma Spectrometer	1. Clean torch assembly and mixing chamber when discolored or after eight hours of running high dissolved solid samples. 2. Clean nebulizer as needed. 3. Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP.	1. Torch and mixing chamber 2. Nebulizer
UV Colorimeter	1. Inspect pump tubes after each 8-hour run; replace if discolored or distorted. 2. Inspect colorimeter daily; replace lamp as necessary.	1. Pump tubes 2. Colorimeter lamp

TABLE 5.3B

ROUTINE PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES
REMEDIAL ACTION/GROUNDWATER TREATMENT
WAUKEGAN MANUFACTURED GAS AND COKE PLANT SITE
WAUKEGAN, ILLINOIS

STAT ANALYSIS CORPORATION

Instrument	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Inductively Coupled Plasma Spectrometer	1. Clean torch assembly and mixing chamber when discolored or after eight hours of running high dissolved solid samples. 2. Clean nebulizer as needed. 3. Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP. 4. Clean cones weekly. 5. Clean lenses every six months, or when needed. 6. Monitor EM voltages and replace EM when needed.	1. Torch and mixing chamber 2. Nebulizer 3. Sample and skimmer cones 4. Mixing T's
Cold Vapor Mercury Analyzer	1. Clean quartz window as necessary 2. Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP. 3. Check tubing daily and replace as necessary 4. Clean Gas-liquid separator.	1. Mercury lamp 2. Tubing 3. Gas-liquid-separators
UV Colorimeter	1. Inspect pump tubes after each 8-hour run; replace if discolored or distorted. 2. Inspect colorimeter daily; replace lamp as necessary.	1. Pump tubes 2. Colorimeter lamp

Attachment 1

Laboratory Standard Operating Procedures

Part A

Environmental Monitoring Technologies, Inc. Standard Operating Procedures

TABLE OF CONTENTS

TITLE: AMMONIA (TITRIMETRIC WITH DISTILLATION)

KEY WORDS: Not applicable.

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY: Matt Gregory /Mary Lubitov August 23, 2007

APPROVALS:

WET. LAB. GROUP LEADER: August 23, 2007
Matt Gregory

QUALITY ASSURANCE MANAGER: August 23, 2007
Mary Lubitov

AMMONIA (TITRIMETRIC WITH DISTILLATION)

1.0 SCOPE AND APPLICATION

1.1 The method covers the determination of ammonia-nitrogen in liquid samples including: drinking waters, surface run-off waters, domestic waters, and industrial waste samples.

1.2 Ammonia occurs naturally in waste and surface waters as part of the nitrogen cycle where it is generally produced by de-amination of organic nitrogen compounds. Ammonia is used in treatment plants to form chlorine residuals. High levels of ammonia in the environment can also have toxic affects on various organisms.

2.0 SUMMARY OF METHOD

The sample is buffered to pH 9.5 with borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds and distilled into a solution of boric acid, which traps the ammonia coming off the distillate. The ammonia present in the distillate is determined titrimetrically with standardized H_2SO_4 and mixed indicator solution.

3.0 DEFINITIONS

Refer to EMT "Quality Manual" for definition of terms used in this SOP.

4.0 SAFETY

4.1 Care needs to be taken while making up dilutions of NaOH as solutions will get hot. Pyrex or equivalent glassware should be used to make the solutions.

4.2 *All samples handled should be considered hazardous and the appropriate PPE should be worn when running this test (safety glasses, gloves, and a lab coat).*

4.3 *Each chemical compound used in this method should be treated as a potential health hazard. MSDS for chemicals are kept at the front desk of 8100 N Austin building.*

4.4 *The use of laboratory equipment and chemicals exposes the analyst to several potential hazards and good laboratory technique and safety practices should be practiced at all times including the use of safety glasses, laboratory coats and acid resistant gloves when handling samples or reagents, or when in the vicinity of others handling these items.*

4.5 *Spilled samples and reagents should be cleaned up from laboratory surfaces immediately. Acidic and Alkaline spills should use spill kit "pigs" for caustic spills.*

4.6 *All additional company safety practices and procedures should be followed at all times.*

4.7 *Also refer to Chemical Hygiene plan in 8100 Conference room.*

NOTE: Please refer to the latest version of EMT' s Chemical Hygiene Plan (CHP) for more comprehensive and authoritative safety information. The information provided in this section is to be used as guidance. The information given in the CHP supercedes the information provided here.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Maximum holding time is 28 days at 4 °C prior to analysis. Samples are preserved with H_2SO_4 to pH < 2.

5.2 Samples to remain in login or wastewater cooler at 4 °C until analysis.

6.0 INTERFERENCE

6.1 Glycine, urea, glutamic acid, cyanates and acetamide hydrolyze very slowly in solution but of these, only urea and cyanates will hydrolyze on distillation at pH of 9.5.

6.2 Volatile alkaline compounds if present (such as hydrazine and amines) may also contribute to the final titrimetric results.

6.3 Residual chlorine reacts with ammonia. If sample is likely to contain residual chlorine, it should be de-chlorinated by addition of $Na_2S_2O_3$ at the time of collection. 0.2mL of 0.35% Sodium Thiosulfate is needed to remove 1mg/L residual chlorine in 100mL.

7.0 EQUIPMENT AND SUPPLIES

7.1 Burette 50-mL

7.2 Distillation apparatus:

7.2.1 Connecting tube

7.2.2 Erlenmeyer (receiving) flask 200-mL

7.2.3 Graham condenser

7.2.4 Kjeldahl flask 500-mL; 800-mL.

7.3 Glass stirring rod

7.4 Heater

7.5 pH paper Narrow range, pH 6.5-10.0

7.6 Volumetric flasks, Class A. 500mL and 1 L.

7.7 Volumetric pipettes, Class A.

8.0 REAGENTS AND STANDARDS

8.1 ***Reagents and Standards Labeling Requirements.***

8.1.1 *Container of commercially bought standard and reagent must have label with Standard/Reagent Name, Initial Concentration, Manufacturer, Lot Number, Expiration Date, Date Received, and Date Opened.*

8.1.2 *Container of prepared solution must have label with Name of Solution, Concentration, Expiration Date and Traceability to the preparation.*

8.1.3 *Reagents and Standards Log Book must have the following information: Standard/Reagent Name, Manufacturer, Lot Number, Date Opened, Expiration Date, Initial Concentration, Initial*

Volume, Final Volume, Final Concentration, Initials of Person received and prepared the solution.

NOTE: *Follow the expiration date listed on the stock reagent or listed below by prepared reagent. Re-order reagent and stock solutions as needed.*

8.2 Ammonia-Nitrogen Certified Standard. Store per manufacturer instruction. Use for LCS preparation.

8.3 Borate Buffer solution: Add 88mL 0.1 N NaOH to a solution of 4.75g sodium tetra borate- $Na_2B_4O_7$ (9.5 g of sodium tetra borate ten hydrate-$Na_2B_4O_7x10H_2O$) in 500mL DI water in a 1 L volumetric flask and dilute to mark. Solution is good for 6 months.

8.4 Boric Acid solution: Dissolve 20 g boric acid (H_3BO_3) in D.I. water and dilute to mark in 1 L volumetric flask. Solution is good for 6 months.

8.5 DI water, ASTM Type II.

8.6 Mixed Indicator solution: Dissolve 100mg methyl red in 50mL 95% ethyl or isopropyl alcohol or methanol. Dissolve 100mg methylene blue in 50mL 5% ethyl or isopropyl alcohol. Combine solutions. Prepare fresh every month.

8.7 Phenolphthalein Indicator Solution: Dissolve 0.5g of phenolphthalein in 50mL of methanol or isopropanol. Solution is good for 6 months.

8.8 Sodium Carbonate (Na_2CO_3)

8.9 Sodium Hydroxide (NaOH), 1N: Dissolve 40g NaOH in D. I. water and dilute to 1L. Holding time is 6 months.

8.10 Sodium Hydroxide (NaOH), 6N: Dissolve 240g NaOH in D.I. water and dilute to 1L. Holding time is 6 months. (Use caution when making the 6 and 1 N NaOH solutions, as the solution will get hot)

8.11 Stock Ammonia solution: commercially prepared, at 1000ppm.

8.12 Sulfuric Acid titrant: 0.02 N H_2SO_4 1mL = 0.28 mg NH3-N. Ready to use, commercially available. Standardize every lot (see below for standardizing information). Refer to expiration date on bottle from manufacturer.

8.13 Tris (hydroxymethyl)-Aminomethane

9.0 CALIBRATION AND STANDARDIZATION

9.1 Standardization of titrant 0.02 N H_2SO_4 with Sodium Carbonate:

9.1.1 Transfer 2 to 4g of sodium carbonate (Na_2CO_3) to crucible and dry at 250 °C for 4 hours. Cool in desiccator and store in tightly sealed container.

9.1.2 Weigh out 0.1060 +/- 0.0100g of the dried Na_2CO_3 and dissolve with DI water in a 100-mL volumetric flask and dilute to mark.

9.1.3 Transfer 10.0mL of Na_2CO_3 solution to 250-300mL conical flask; add 50mL of water and 2 drops of a 0.1% solution of methyl red in isopropyl alcohol.

9.1.4 Titrate with the 0.02 M H_2SO_4 solution to the first appearance of a red color and boil the solution carefully (to avoid loss) until the red color disappears then cool to room temperature.

9.1.5 Continue the procedure in step 4 until the red color does not fade during the heating process.

9.1.6 Calculate the normality of the H_2SO_4 solutions, see Sec.11.1 for calculation. (Duplicate titrations must be run and must agree within 0.0004 normality units to be accepted. If the normality of solution is found to lie outside the range 0.0196-0.204 N, the normality determined by standardization should be used in calculations).

9.2 Standardization with Tris (hydroxymethyl)-Aminomethane:

9.2.1 Dry 10g of Tris (hydroxymethyl)-Aminomethane at 70 °C for 24 hours. Cool in a desiccator.

9.2.2 Weigh 0.04 g of Tris (hydroxymethyl)-Aminomethane in 250-mL beaker and dissolve in 50 mL of DI water.

9.2.3 Titrate Tris (hydroxymethyl)-Aminomethane solution with 0.02 N H2SO4 to pH 4.5 using a pH meter.

9.2.4 Calculate the normality of the H_2SO_4 solution, see Sec.11.2 for calculation.

9.2.5 Duplicate titrations must be run. Results that agree within 0.0004 normality units are acceptable for averaging. The exact normality is to be used in calculations.

10.0 PROCEDURE

10.1 Preparation of equipment:

10.1.1 Add to the Kjeldahl flask ~ 200mL of DI water, 25mL borate buffer, 3-4mL 1N NaOH and boiling chips. Steam out the apparatus by distilling approximately 150mL of distillate.

10.1.2 Leave the apparatus assembled after cleaning until required. (Samples must be analyzed within four hours after cleaning procedure).

10.1.3 After all titrations have been performed, empty the burette and rinse titrant out thoroughly with DI water. Keep burette in upside down position protected from dust.

10.2 Liquid samples:

10.2.1 Measure 200mL of wastewater to the Kjeldahl flask. Use smaller aliquot if the sample is known to be highly contaminated. Add 3 drops of phenolphthalein and 6N NaOH till solution turns pink. Make final pH adjustment to 9.5 +/- 0.5 using 0.1N NaOH, under the control of pH paper.

10.2.2 Add 20mL of the borate buffer.

10.2.3 Add sufficient D.I. water to give a volume of approximately 250mL in the distillation flask.

10.2.4 To a 200-mL Erlenmeyer flask add 50mL boric acid solution and approximately 6 drops of the mixed indicator solution.

10.2.5 Position the Erlenmeyer flask so that the outlet tube from the condenser is under the surface of the boric acid solution and will remain immersed in the solution during the distillation. Be sure to turn on water supply to the condensers prior to the start of heating the samples or a "backflow" may occur in which that sample (or batch) will need to be re-set.

10.2.6 Distill at a rate of 6 to 10mL/min. Collect between 150 and 200mL of distillate. Presence of ammonia is indicated by the boric acid solution turning green.

10.2.7 Titrate the sample distillates collected in the Erlenmeyer flasks with 0.02 N sulfuric acid, using the blank end-point color as reference.

10.2.8 Carry a blank through all steps of the procedure and apply the necessary correction to the result by using the blank flask color as the end-point for the titration.

10.3 Sludge or solid samples:

10.3.1 Weigh 2-3g of sample in a plastic beaker, wash the sample into Kjeldahl flask with ~ 200mL of DI water, adjust pH with 1 N NaOH to 9.5 +/- 0.5 under the control of pH paper. Add 20mL of borate buffer. Less sample may be used for solid if high level of ammonia suspect. Reporting limit must be adjusted for sample size by reporting less than MDL X Dilution Factor if sample turns out to be low.

10.3.2 Proceed with the procedure as for liquid samples from step 10.2.3.

10.3.3 If sample requires more than 50mL of titrant, transfer the sample to a 250-mL volumetric flask and bring the volume to the mark with DI water. Use 50mL of the diluted sample for titration.

11.0 CALCULATIONS

11.1 Calculate the normality of the H_2SO_4 solutions as follows:

$$A = \frac{B}{(0.53)\,C}$$

Where:
A = normality of H_2SO_4 solution (0.02 M)
B = grams of NA_2CO_3 used.
C = milliliters of H_2SO_4 solution used until color did not fade.

11.2 Calculate the normality of the H_2SO_4 solution, as follows:

$$A = B / 0.1211 * C$$

Where:
A = normality of the H_2SO_4 solution
B = grams of Tris (hydroxymethyl)-Aminomethane used
C = mL of H_2SO_4 consumed.

11.3 For Ammonia as N:

$$\text{mg/L NH3-N} = \frac{(A-B) \times 14000 \times 0.02N}{S}$$

Where:
S = mL (or g if solid) of sample
A = mL sulfuric acid used for sample
B = mL sulfuric acid used for blank
14000 = mass of N
0.02N = normality of sulfuric acid

11.4 Calculate mg/L NH3-N and multiply by DF.

DF= total volume of diluted sample/ volume of titrated aliquot.

11.5 True Value (TV) of Spike mg/L =

$$\frac{\text{(mL of spiking solution) x (concentration of spiking solution)}}{\text{Volume of Sample}}$$

11.6 Matrix Spike Recovery % =

$$\frac{\text{MS mg/L} - \text{Sample mg/L (if greater than MDL)} \times 100}{\text{TV of Spike}}$$

11.7 *Calculation of the common quality control parameters (e.g., percent recovery, percent difference, relative percent difference (RPD), and relative standard deviation (RSD)), are given in the EMT Laboratory Quality Assurance Manual.*

12.0 QUALITY CONTROL

12.1 Run **Method Blank** with each analytical batch (batch = number of samples in a 24 hour period if less than 20) or every 20 samples. The method blank (200mL of D.I. water) is subjected to the same steps of procedure as a sample. Samples associated with the contaminated method blank should be reanalyzed. Only the results less than MDL or the results 10 (for liquids) to 20 (for solids) times higher than MDL can be reported. The source of the method blank contamination should be located and eliminated.

12.2 **Laboratory Control Sample (LCS)** should be analyzed with every batch. LCS is prepared from the certified source standard. Acceptable LCS recovery is 90 -110%. Batches with failed LCS need to be reset.

12.3 **Matrix spike and matrix spike duplicate (MS/MSD)** should be analyzed at frequency one per 20 samples. If sample is expected to have high level of ammonia, analyze duplicate instead of matrix spike duplicate. To be acceptable MS/MSD recovery should be within 80 - 120% and RPD <20%. The failure to meet recovery or RPD limits needs to be investigated. In some instances the samples or the whole batch might need to be re-analyzed. The results associated with the failed MS/MSD or RPD must be flagged.

12.4 Any non-standard situations, problems and corrective actions need to be documented.

12.5 After 15 to 20 analyses generate **Laboratory Control Limits** for LCS and MS/MDS recovery and RPD. Update the control limits semiannually or after 25-30 additional data points, whichever is less frequent. Use laboratory control limits for data validation if they are more stringent than the pre-established limits. Regularly (every 10-20 points) review control charts for indication of systematic trends.

13.0 REPORTING

For reporting refer to the most current revision of SOP # 209.

14.0 DEVIATIONS FROM THE REFERENCE METHOD

14.1 Distillation apparatus is steamed out with addition of NaOH and borate buffer to guarantee better cleaning efficiency.

14.2 Standardization of 0.02 N H2SO4 with Tris (hydroxymethyl)-Aminomethane is suggested as an alternate method to standardization versus sodium carbonate.

15.0 METHOD PERFORMANCE

15.1 Initial Demonstration of Capability (IDC) must be performed when a new analyst begins to work or the analytical procedure is changed. Four aliquots of at level 10-12 times MDL should be processed through the entire procedure. Recovery for all replicates should fall within 90-110%.

15.2 MDL study or MDL verification should be done annually following the most recent revision of SOP # 218.

15.3 *Method performance is detailed in the EMT Quality Assurance Manual, which includes sections on Method Startup, Reporting Limits, Method Detection Limits (MDLs), Method Control, and Initial Demonstrations of Capability (IDC).*

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 Aqueous sample waste may be disposed of into a sink designated for liquid waste and the reagents into the proper waste containers.

16.2 Prepare standards and reagents in volumes consistent with the laboratory use to minimize the disposal of excess of expired standards and reagents.

16.3 *Refer to EMT "Quality Manual" for samples disposal used in this SOP.*

17.0 TABLES, DIAGRAMS, FLOW CHARTS, INSTRUMENT MAINTENANCE

Not applicable.

18.0 REFERENCES

18.1 *Laboratory Quality Assurance Manual, Environmental Monitoring & Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.*

18.2 *Standard Methods for Examination of Water and Wastewater. 18th Edition. Method 4500 NH_3E.*

PHENOLICS SPECTROPHOTOMETRIC, 4-AAP WITH DISTILLATION

Scope and application: This method is applicable to the analyses of drinking water, wastewater, domestic and industrial waste.

Summary: Phenolics materials react with 4-aminoantipyrine in the presence of Potassium ferricyanide at pH of 10 to form a stable reddish- brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolics material.

Samples handling and preservation:
The samples are collected in glass containers and preserved by acidification to a pH <2 with H_2SO_4 and kept at 4oC.The maximum holding time is 28 days.

Interferences:
Phenol-decomposing bacteria reduce phenol concentration in water. Biological degradation is inhibited by the addition of 1 g per 1L of $CuSO_4$ and acidification.

Oxidizing Agents may oxidize some or all of the phenols in a short time. Ferrous sulfate added to the samples will destroy oxidizing substances.

Sulfur compounds that liberate hydrogen sulfide or sulfur dioxide (SO2) on acidification may interfere with the phenol determination. This interference is eliminated by addition of sufficient quantity of $CuSO_4$ to the sample before distillation and adjusting pH of sample to < 4.

The pH 4 is critical to the success of distillation. Most of the recoverable phenols will be in the acid form at pH 4, and the other non-reactive phenols, such as nitrophenols will be in the base forms and not be recovered in the distillate.

Some distilled substances can create turbidity in the sample. Prepare sample blank with all reagents, except 4-AAP, to correct for sample turbidity.

Apparatus:
- Spectrophotometer: HACH 4000 or equivalent.
- Distillation apparatus: A 1 L distilling flask attached to a condenser. Clean condensers by rinsing with DI water and acetone.
- Boiling chips
- pH Test paper in range 9.2-10.6.
- Class A volumetric flasks 1000, 500, 250 mL.
- Class A volumetric pipettes 50 mL, 10 mL.
- Assorted calibrated pipetters
- Erlenmeyer flasks 250 and 500 mL.
- Class B graduated cylinders 250, 50 mL.
- Cuvette 10 and 50 mm
- Analytical balance capable of weighing to 0.01g
- Analytical balance capable of weighing to 0.1g

Reagents:
All reagents used in the test should be ACS or higher grade.
- Sulfuric acid H_2SO_4 18N: Add slowly 500 mL of conc. H_2SO_4 to 500 mL of D.I. water.
- Sulfuric acid H_2SO_4 10%: Add slowly 25 mL of conc. H_2SO_4 to 250 mL of D.I. water.
- Sodium Hydroxide (NaOH) 10% w/v: Dissolve 25 g of NaOH in 250 ml of water
- Copper sulfate solution: dissolve 100 g $CuSO_4.5H_2O$ in D.I. water and dilute to 1L.
- Buffer solution: Dissolve 16.9 g NH_4Cl in 143 mL conc. NH_4OH and dilute to 250 mL with distilled water. 1 mL should adjust 50 mL of distillate to pH 10.
- Aminoantipyrine solution: dissolve 2 g 4AAP in D.I. water and dilute to 100 mL. Prepare daily.
- Potassium ferricyanide: dissolve 8 g of $K_3Fe(CN)_6$ in D.I. water and dilute to 100 mL. Prepare fresh weekly.
- Bromate-Bromide solution: Dissolve 2.784 g anhydrous $KBrO_3$ in D.I. water, add 10 g KBr crystals, dissolve, and dilute to 1000 mL.
- Starch solution (1%): commercially purchased.
- Standard sodium thiosulfate titrant ($Na_2S_2O_3$) 0.025 N: commercially purchased and restandardized

monthly after opening.

- KI, crystals
- Stock phenol standard 1000 mg/L. Dissolve 1.0 g phenol in freshly boiled and cooled D.I.water and dilute to 1 L. Prepare fresh stock solution monthly and standardize. ***This solution is toxic.***
- Intermediate phenol standard 50 mg/L: dilute 5 mL of 1000 mg/L standard to 100 with D.I. water. Prepare weekly.
- Intermediate phenol standard 2.5 mg/L: dilute 0.25 mL of 1000 mg/L standard to 100 with D.I. water. Prepare daily.
- Certified Phenol standard 1000 mg/L. Store per manufacturer recommendations.
- Methyl Orange Indicator Solution: Dissolve 0.1 g of methyl orange in 100 mL of water and filter if necessary.
- ASTM type II reagent water

Standardization procedure for 1000 mg/L phenol standard prepared in the laboratory:
Transfer 100 mL D.I. water in a 500 mL glass stoppered conical flask, add 50.0 mL stock phenol solution and 10 mL of bromate-bromide solution.
Immediately add 5 mL conc. HCl and swirl gently. If brown color of free bromine does not persist add 10.0-mL portions of bromate-bromide solution until it does. Usually four 10-mL
portions of bromate-bromide solution as required.
Let stoppered flask stand for 10 min, than add ~ 1 g KI.
Prepare blank, 100 mL of D.I. water, and treat it in exactly the same manner.
Titrate blank and standard solution with 0.025 N $Na_2S_2O_3$ using starch solution as indicator.

Calculate the concentration of phenol solution:
mg phenol/L = 7.842[(AxB) - C]
Where:
A= mL $Na_2S_2O_3$ used for blank

B= mL bromate-bromide solution used for sample divided by 10
C = mL $Na_2S_2O_3$ used for phenol stock solution

Duplicate titration must be run and must agree within 0.5 mL to be accepted. If the concentration of solution is found to lie outside the range 999.5 -1000.5 mg/L, the concentration determined by standardization should be used in calculations.

Definitions:
There are not definitions unique to this method.

Pollution prevention:
Prepare standards and reagents in volumes consistent with the laboratory use to minimize the disposal of excess of expired standards and reagents.

Waste Management:
Dispose distilled samples down the sink for disposal of wastewater samples. Expired standard should be collected in the CN waste container.

Safety:
Each chemical compound used in this method should be treated as a potential health hazard. MSDS for chemicals are kept near the front desk in 8100 N Austin building.

PROCEDURE:

Distillation:

1. For liquid samples: place 250 mL of sample in a round bottom 1 L flask.
2. For solid samples: place 10 g and 250 mL D. I. water in a 1 L round bottom flask.
3. Adjust the pH of the solution in the flask to approximately 4 with 18N H_2SO_4 or 10% NaOH, using the methyl orange indicator.
4. Add 2.5 mL of the copper sulfate solution.
5. Heat the flask and distill over, into a clean weighed collection vessel, 200 mL of liquid.
6. Turn off the heat for 10 minutes and then add 50 mL of D.I. water to the distillation flask.
7. Turn on the heat again and distill over an additional 50 mL. Upon distillation completion weigh the collection vessel with distillate. The total amount of distillate = Wt of vessel with distillate – Wt of empty vessel. Record all weighs.

. **Colorimetry:**

1. Allow the HACH 4000 to warm up for 30 minutes. Recall calibration method.
2. Filter the distillate if it is cloudy or if it has sediment.
3. Measure 50 mL of the distillate (or an aliquot made up to 50 mL) into a 50 ml plastic cup and adjust the pH to 10 by adding 1 mL of buffer solution. The pH should be 10+/-0.2.
4. Add 1.0 mL of 4-aminoantipyrine solution and mix.
5. Add 1.0 mL of potassium ferricyanide solution
6. Allow 15 minutes for the color to fully develop.
7. Pour sample in the cuvette. Use 10 mm cuvette for regular level and 50 mm cuvette for low-level phenolics.
8. Prepare calibration blank using 50 ml of D.I. water and following steps
9. Zero the instrument using calibration blank.
10. Read the samples. If the reading is above calibration range, repeat colorimetrical determination with smaller volume of distillate diluted to 50 ml. Record in the bench book the sequential number of measurement against the sample number. Save the electronic data produced by HACH in the directory: Data 1 on Ralph (L:)\Wc\HACH 4000\ year-month. Give the reference to the file name in the bench book.

Calculation:

$$\text{phenol ppm} = \frac{A \times (\text{vol. distillate collected}) \times DF}{B}$$

Where:

A = ppm phenol as read from curve
B = mL or g of original sample taken
DF = 50/V; V= mL of distillate used for analysis

Calibration:

Prepare calibration blank and minimum 5 calibration standards covering the working range of measurement. The lowest standard should be at reporting limits. The calibration standards should be evenly distributed with the calibration range. Prepare calibration standards from the certified stock standard solution or freshly made and standardized 1000 mg/L laboratory standard.
Example of calibration curve preparation for regular level phenol

	Initial Concentration mg/L	Initial volume mL	Final volume mL	Final concentration mg/l
Calibration Blank	---	-----	50	0.0
Standard 1	50	0.1	50	0.1
Standard 2	50	1	50	1.0
Standard 3	50	2	50	2.0
Standard 4	50	3	50	3.0
Standard 5	50	4	50	4.0

Example of calibration curve preparation for low level phenol

	Initial Concentration mg/L	Initial volume mL	Final volume mL	Final concentration mg/l
Calibration Blank	---	----	50	0.0
Standard 1	2.5	0.1	50	0.005
Standard 2	2.5	1	50	0.05
Standard 3	2.5	3	50	0.15
Standard 4	2.5	5	50	0.25
Standard 5	2.5	8	50	0.40

Process calibration standards as samples following steps 3 through 8 from "Colorimetry" part. Adjust wavelength to 510 nm. Zero the instrument with calibration blank and read the absorbance of the standards.
Use **10 mm** cuvette for reading of regular level phenol and **50 mm** cuvette for reading of low-level phenolics.

Open the "User Programs" entry and create a new program. Plot absorbance values versus concentration. Coefficient of correlation should be > 0.995 to consider calibration curve acceptable. If the coefficient of correlation < 0.995 prepare new calibration. Save the calibration under directory: L:\Wc\HACH 4000.
Verify calibration with the 1 mg/L check standard from the secondary source. Acceptable range for check standard recovery is 90-110%.
Update calibration curve once a year or with every new lot of reagents.

Quality Control

Daily Spectrophotometer check:

Prepare calibration blank to zero the instrument.
Verify calibration curve by analyzing check standard in the beginning of the run, at the end of the run and every 15 measurements. Use 1 mg/L check standard for regular range verification and 0.05 mg/L for low range. Acceptable range for check standard recovery is 90-110%.

Check standard preparation:

	Initial Concentration mg/L	Initial volume mL	Final volume mL	Final concentration mg/l
Check standard low range	50	0.1	100	0.05
Check standard regular range	50	2	100	1

If the recovery is outside of the limits, prepare fresh standard and repeat verification. If the standard is still outside of 90-110 %, prepare new calibration curve.

Batch QC:

1. Batch QC samples should be brought through the whole sample preparation and analytical process.
2. Analyze Method blank with every 20 samples or once per batch. Method blank should be less than MDL. If the Method Blank value exceeds MDL level investigate the problem and reanalyze the batch. Only the samples with phenolics concentration less than MDL or greater than 10x Method Blank value can be reported
3. Analyze LCS (Laboratory Control Sample) with every batch or 20 samples.
   Prepare regular range LCS by spiking 250 mL of DI water with 0.1 ml of 1000 mg/L phenol standard. The target value of LCS is 0.4 mg/L.
   Prepare range LCS by spiking 250 mL of DI water with 0.25 ml of 50 mg/L phenol standard. The target value of LCS is 0.05 mg/L.
   The recovery of LCS should be within the laboratory control limits or 90-110 % whichever is more stringent. If LCS is outside of the acceptance range investigate the problem. The samples associated with failed LCS must be reanalyzed.
4. Analyze one MS/MSD (matrix spike/ matrix spike duplicate) per every 10 samples. The level of spike should be at least 10-20 times MDL or 1/2 of sample value whichever is higher. Use LCS level if the concentration of phenol in the sample is unknown. The recovery of MS/MSD should be within the laboratory control limits or within 80-120 % whichever is more stringent. The RPD should be within the laboratory RPD limit or <20% whichever is more stringent. Investigate the problem if RPD exceed control limits or the recovery of MS/MSD is outside of acceptance range:
5. Check calculations
6. Repeat colorimetric test with smaller volume of distillate
7. Repeat distillation and colorimetric determination using smaller sample size. If problem cannot be resolved, flag the data. The problem is considered matrix related if the associated LCS meet the acceptance criteria.
8. Generate control limits for % recovery of LCS, MS/MSD and RPD. Use the limits generated in the laboratory for the data evaluation if they are more stringent than the pre-set limits. Update control limits semiannually. Check control charts for systematic trends at least monthly.
9. Initial Demonstration of Capability (IDC) must be performed when a new analyst begins to work or the analytical procedure is changed. Prepare four standard with concentration approximately 10 to 20 times

MDL and processed through the entire procedure. The standards should be from a source different from the source of the calibration standards. The study is acceptable when recovery for all 4 replicates fall within 90-110%.

10. Document encountered problems, corrective measures and the efficiently of corrective action as required per SOP # 247 rev 0.
11. Method Detection Limit (MDL) should be established and verified annually. Determine MDL following SOP # 218 rev.1.

Deviations from the referenced methods EPA method 420.1 or SW-846 method 9065.

1. The aliquot used for distillation and the volume of distillate are reduced to 250 ml versus 500 ml in the 420.1 and 9065.
2. The aliquot used in photometric determination is reduced 50 mL versus 100 ml in the methods. The volume of all reagents used in colorimetric determination is adjusted proportionally.
3. Samples preservation and pH adjustment are done with sulfuric acid per 9065 and 40 CFR Ch.1. EPA method 420.1 suggests to use phosphoric acid.
4. The modifications to the procedure are done to cut down the amount of waste. The modifications do not affect adversely the sensitivity of the method or quality of the data. The laboratory MDL is 20 ug/L versus 50 ug/L stated in the EPA method; the LCS (0.4mg/L) recovery is within 90-110%.

Data Reporting:

The MDL and PQL in OMEGA are based on aliquot size 250 ml or g for liquids and for soils. The MDL and PQL adjustment per aliquot size and final volume of distillate is done by the OMEGA. Refer to SOP # 261.

Reference:

EPA SW-846 Method 9065
EPA MCAWW Method 420.1

Revised by: E. Talkovska 08/24/04
Approved by: Mitchell Ostrowski 12/27/04

TITLE: METHOD 3015, MICROWAVE DIGESTION FOR AQUEOUS SAMPLES AND EXTRACTS

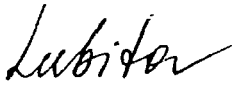
KEY WORDS: Not applicable.

COMMENTS: The SOP was revised to convert to a new format and to incorporate the MARS5 microwave operation guide. *Italicized items indicate changes from the last revision.*

REVISED BY: Mioara Bratian August 23, 2007

APPROVALS:

METAL GROUP LEADER:  August 23, 2007
Mioara Bratian

QUALITY ASSURANCE MANAGER:  August 23, 2007
Mary Lubitov

METHOD 3015, MICROWAVE DIGESTION FOR AQUEOUS SAMPLES AND EXTRACTS

1.0 SCOPE AND APPLICATION

This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts (from TCLP, SESW, etc.) and wastes that contain suspended solids for analysis, *by ICP-MS* and ICP-AES. This method involves a microwave digestion of the sample with Nitric acid (HNO_3).

2.0 SUMMARY OF METHOD

Nitric acid is added to an aqueous sample in a 120-mL Teflon digestion vessel. The vessel is capped and heated in a microwave unit. After cooling, the contents are filtered, centrifuged, or allowed to settle in a clean sample bottle for the eventual analysis of the metals content.

3.0 DEFINITIONS

Refer to EMT "Quality Manual" for definitions of terms. No definitions unique for the test.

4.0 SAFETY

4.1 Always add the concentrated acid slowly to the water (not water to acid) to avoid a reaction.

4.2 Microwave unit needs to be connected to a hood to remove any vapors that may escape during digestion. Addition of acid to samples should be carried out in a fume hood in case toxic nitrous oxide fumes are evolved.

4.3 When opening vessels after digestion, open in hood facing away from operator after contents are at or below room temperature.

4.4 *All samples handled should be considered hazardous and the appropriate PPE should be worn when running this test (safety glasses, gloves, and a lab coat).*

4.5 *Each chemical compound used in this method should be treated as a potential health hazard. MSDS for chemicals are kept at the front desk of 8100 N Austin building. Be aware of the hazardous effects of Hg and the reagents when inhaled or contacted with skin.*

4.6 *The use of laboratory equipment and chemicals exposes the analyst to several potential hazards and good laboratory technique and safety practices should be practiced at all times including the use of safety glasses, laboratory coats and acid resistant gloves when handling samples or reagents, or when in the vicinity of others handling these items.*

4.7 *Spilled samples and reagents should be cleaned up from laboratory surfaces immediately. Acidic and Alkaline spills should use spill kit "pigs" for caustic spills.*

4.8 *All additional company safety practices and procedures should be followed at all times.*

4.9 *Also refer to Chemical Hygiene plan in 8100 Conference room.*

NOTE: Please refer to the latest version of EMT' s Chemical Hygiene Plan (CHP) for more comprehensive and authoritative safety information. The information provided in this section is to be used as guidance. The information given in the CHP supercedes the information provided here.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Samples are to be collected in metal free containers such as glass quart jars or plastic pint jars with plastic or Teflon lids. To delay the chemical and biological changes that inevitably continue after the sample is removed from the parent source, a preservation technique is required. Metal ions may precipitate as hydroxides or form complexes with other constituents. Cations or anions may change valence states under certain reducing or oxidizing conditions while other constituents may dissolve or volatilize with the passage of time. Metal cations may also absorb onto surfaces of the container (glass, plastic, etc.), such as iron and lead. Preserve the liquid samples for metals analysis to a pH < 2 with HNO_3. HNO_3 keeps the metal ions in solution and delays the ionic exchange between the metallic ions and the surface of the container.

5.2 All samples should be preserved as soon as possible after collection and kept on ice if not preserved. After receipt, samples are to be logged in and stored in the login cooler at 4 °C.

5.3 Even though the holding time for metals is 6 months, it is best to analyze the samples as soon as possible after collection.

6.0 INTERFERENCE

6.1 Samples that are very reactive or volatile may create high pressures when heated and may cause venting of the vessels with potential loss of sample and analytes.

6.2 Samples that contain carbonates or other carbon dioxide generating compounds may cause enough pressure to vent the vessel. If this situation is anticipated, use smaller sample amount and dilute with DI water to have the desired 45mL of sample with 5mL of the HNO_3. Vessel weights are checked before and after the digestion so that if a loss greater than 10% occurs, the sample is to be re-digested.

7.0 EQUIPMENT AND SUPPLIES

7.1 Auto pipettes of 1 and 5mL capacities to be calibrated monthly.

7.2 Dispensers for acid and spike solutions to be calibrated with each new standard or change of HNO_3

7.3 Filter paper, Whatman #4 or equivalent. Filters do not require any treatment unless the target analytes are: Al, Co, Mo, V, K, Mg, Na, Ca: soak filters in 10% HNO3 for 4-5 hrs then rinse at least 3 times with ~ 50mL of DI water.

7.4 Fume Hood (face velocity 100 fpm)

7.5 Graduated cylinders, 50-mL capacity

7.6 Microwave ovens: MSP1000 or *MARS 5* by CEM should be calibrated/checked quarterly or more often if needed. Calibration should always be performed after microwave oven repair. Refer to Appendix B, C and D for the calibration procedure.

7.7 Polypropylene filter funnels.

7.8 Teflon Beaker, 1L, for use in microwave calibration.

7.9 Thermometer capable of measuring +/- 0.05 °C for Microwave calibration.

7.10 Top loading balance Scout Pro 400g with +/- 0.01g accuracy. Balance is to be checked/ calibrated weekly according to manufacturer instructions. Used only to weigh digestion vessels against loss during digestion.

7.11 Volumetric flasks (1000, 500, and 250-mL) for the making of spike solutions. Class A.

8.0 REAGENTS AND STANDARDS

8.1 *Reagents and Standards Labeling Requirements.*

8.1.1 *Container of commercially bought standard and reagent must have label with Standard/Reagent Name, Initial Concentration, Manufacturer, Lot Number, Expiration Date, Date Received, and Date Opened.*

8.1.2 *Container of prepared solution must have label with Name of Solution, Concentration, Expiration Date and Traceability to the preparation.*

8.1.3 *Reagents and Standards Log Book must have the following information: Standard/Reagent Name, Manufacturer, Lot Number, Date Opened, Expiration Date, Initial Concentration, Initial Volume, Final Volume, Final Concentration, Initials of Person received and prepared the solution.*

NOTE: *Follow the expiration date listed on the stock reagent or listed below by prepared reagent. Re-order reagent and stock solutions as needed.*

8.2 DI water. High purity water (ASTM type II)

8.3 Instrument grade chemicals shall be used in all tests.

8.4 Nitric Acid, (HNO_3), concentrated (low trace metal grade)

8.5 Stock standard solutions of the metals of interest for the making of spiking solutions for each digestion batch. Refer to Appendix A at the end of the SOP for a complete listing of the standards to be made, elements included in each, expiration dates, lot number creation and labeling, and complete preparation instructions.

9.0 CALIBRATION AND STANDARDIZATION

Microwave ovens: MSP1000 *or MARS 5* should be calibrated/checked quarterly or more often if needed. Calibration should always be performed after microwave oven repair. Refer to Appendix B, C and D for the calibration procedure.

10.0 PROCEDURE

10.1 Cleaning of the digestion vessels before setting up samples:

10.1.1 All digestion vessels should be washed with soap and tap water after each digestion cycle.

10.1.2 Use acetone to eliminate oil or organic residue that does not come off by using soap and water from step

10.1.3 Rinse vessels with DI water after steps 10.1.1 and 10.1.2 complete.

10.1.4 Soak all vessels in 10% HNO3 bath for a minimum of 30 minutes before the next batch run. Soak the vessels overnight at the end of each day. 10% HNO_3 bath should be changed at a minimum of once a month to guard against any contamination.

10.1.5 After highly contaminated samples digestion vessels should be cleaned normally and run one digestion cycle using an acidic solution of 30mL DI H_2O, 10mL conc. HNO_3, and 10mL conc. HCl.

10.1.6 Rinse with DI H_2O three times for the final cleaning after pulling vessels out of acid bath prior to use.

10.1.7 Let dry on rack as much as possible before addition of samples so that initial weight prior to *digestion is not biased due to DI water on outside of vessel.*

10.2 Digestion set-up procedure for samples and Microwave usage:

10.2.1 Measure a 40mL aliquot of a well-shaken sample into a graduated cylinder. Slowly add 5mL of concentrated HNO_3 in the fume hood and dilute to a total volume of 50mL with DI. Use smaller aliquot of a sample if the sample is dirty, has an odor, or appears to have a high viscosity (generally 25mL, 10 to 5mL with very bad samples).

10.2.2 For QC samples, measure a 40mL aliquot of a well shaken sample into a graduated cylinder. Add 5mL of concentrated HNO_3 and 1mL of TM spike solution for the initial spike (MS). Also add 2.5mL Odd Spike if minerals are needed. Prepare spike duplicates (MSD) and blank spikes (LCS) in the same manner. The blank is 45mL of DI water. (This will cover the majority of samples to be run- see note below for others).

NOTE: Refer also to Appendix A at the end of SOP for all spiking solutions available and amount of each solution to be added to the sample. With sample and spike solutions, volume of sample plus spikes cannot exceed 45mL due to the addition of 5mL acid. All metals of interest for all samples in the batch must be included in the MS, MSD, and LCS.

10.2.3 Pour the diluted samples (including spikes and acid) to a dried Teflon PFA vessel and cap the vessel. Check to make sure the pressure relief disks are in the caps with the smooth side toward the sample and start the caps a few turns on the vessels.

10.2.4 Finish tightening the caps in the capping station that will tighten them to a uniform torque pressure of 12 ft-lbs. Weigh each capped vessel to the nearest 0.01g and record the initial weight in the digestion book along with volume of sample used, acid lot number used, all lot numbers from added spike solutions, date of digestion, and preparer's initials. *If the digestion will be performed with MARS 5, one vessel should have attached a double port cap and sealing ring.*

10.2.5 Place digestion vessels evenly in the carousel and add a balancing blank to load the carousel evenly if necessary.

10.2.6 Program the microwave unit according to the operation procedure and record in digestion book the power levels from program 1 and 2 that were used (dependent upon the number of vessels in the batch as to the power setting required for thorough digestion and heating of samples to occur).

NOTE: Be sure to turn the hood on when operating the microwave.

10.3 MSP 1000 Microwave operation

10.3.1 Switch on power switch located on the back panel and let unit go through diagnostic tests. (May need to press F1 and F3 quickly to get out of vapor detection mode).

10.3.2 Press F3 to load program.

10.3.3 Press F1 to recall stored method.

10.3.4 Select "Wastewaters" and hit enter key.

10.3.5 Press F1 to load method.

10.3.6 Press F3 to review method.

10.3.7 Set power for program one and two (see chart at end of SOP). Time should be 10 minutes for both (1000 on display).

10.3.8 Press F3 to end.

10.3.9 Press F4 to start digestion.

NOTE: If any problems are incurred, refer to Trouble-Shooting in manufacturer's manual or ask supervisor.

10.4 ***MARS 5 Microwave operation***

10.4.1 *Place the turntable into the microwave. A microwave transparent temperature probe is inserted into the Teflon coated thermo well of a sample vessel and connected to a snap-in port in the center of the roof of the instrument cavity.*

CAUTION: *The RTP-300 Plus probe and the thermo well are both fragile. Exercise care when handling either of them.*

10.4.2 *Switch on power switch located on the panel and let unit go through diagnostic tests.*

10.4.3 *Using the arrow keys, highlight "Edit/Create method". Press the "SELECT" key.*

10.4.4 *Using the arrow keys, highlight "User Directory". Press the select keys.*

10.4.5 *Using the arrow keys, highlight the method.*

10.4.6 *From the "Select Vessel" Type screen, PFA need to be highlighted. If is not, using the arrow keys, highlight the PFA. Press the SELECT key.*

10.4.7 *From the "Select Control type screen", "RAMP TO TEMPERATURE" needs to be highlighted. If not, using the arrow keys, highlight the "RAMP TO TEMPERATURE". Press the SELECT key.*

10.4.8 *From the "Enter method parameters" screen, using the arrow keys, highlight the desired % power. Press the SELECT key twice.*

10.4.9 *Press START.*

NOTE: *If any problems are incurred, refer to Trouble-Shooting in manufacturer's manual or ask supervisor.*

10.5 Final steps of samples preparation after digestion:

10.5.1 After the digestion programs are complete, cool the digestion vessels to room temperature (leave in microwave for about 10 minutes to let cool and allow any venting which may occur). Vessels may be put into cooler to bring temperature down faster.

10.5.2 Weigh and record the weight of each vessel assembly. If the weight of the sample plus acid has decreased by more than 10%, discard the sample and re-digest.

10.5.3 Un-torque the vessels in the capping station and unscrew them by hand. Filter the samples with paper if cloudy or have precipitation into clean, labeled containers.

11.0 CALCULATIONS

11.1 *Calculation of the common quality control parameters (e.g., percent recovery, percent difference, relative percent difference (RPD), and relative standard deviation (RSD)), are given in the EMT Laboratory Quality Assurance Manual.*

11.2 Determinative calculations that are not applicable to the digestion procedure.

12.0 QUALITY CONTROL

12.1 **Laboratory Control Samples (LCSs)** – A laboratory control sample must be prepared and analyzed with every preparation batch of twenty (20) or less samples. Measure a 40mL of DI water into a graduated cylinder. Add 5mL of concentrated HNO_3 and 1mL of **TM** spike solution for the initial spike (MS). Also add 2.5mL Odd Spike if minerals are needed. Dilute to 50mL.

12.2 **Matrix spike/Matrix Spike duplicates (MS/MSD)** – A matrix spike and matrix spike duplicate sample must be prepared and analyzed with every preparation batch of twenty (20) or less samples. Measure a 40mL aliquot of a well shaken sample into a graduated cylinder. Add 5mL of concentrated HNO_3 and 1mL of TM spike solution for the initial spike (MS). Also add 2.5mL Odd Spike if minerals are needed. Prepare spike duplicates (MSD) in the same manner.

12.3 **Method Blank (MB)** – A method blank must be prepared and analyzed with every preparation batch of twenty (20) or less samples. Method blanks are prepared using the same reagents that are used to digest samples, and is carried throughout the entire preparation and analytical process. The blank is 45mL of DI water.

NOTE: Refer also to Appendix A at the end of SOP for all spiking solutions available and amount of each solution to be added to the sample. With sample and spike solutions, volume of sample plus spikes cannot exceed 45mL due to the addition of 5mL acid. All metals of interest for all samples in the batch must be included in the MS, MSD, and LCS).

12.4 The initial **demonstration of Capability (IDC)** is used to characterize instrument performance and laboratory performance prior to analyses conducted by this method. Analysts new to running the method need to perform initial demonstration as soon as possible after training.

12.5 To ensure QC per every 20 samples when only 12 are digested at a time due to carousel size constraints the first carousel is set up with the MBLK and LCS plus ten samples and the second carousel is set up with the MS and MSD plus the remaining 10 samples for a full batch.

13.0 REPORTING

For reporting refer to the most current revision of SOP # 209.

14.0 DEVIATIONS FROM THE REFERENCE METHOD

14.1 Deviations from the referenced method SW-846 3015:

14.2 Filters are not treated with HNO3 unless the target analytes are Al, Co, Mo, V, K, Mg, Na, and Ca. The accumulated method blank data shows that the filters do not contain other traces of metals besides listed above. The filter treatment might be changed if the contamination problem traced to the filters arises.

14.3 Calibration of MW MPS 1000 is done at working range 20 to 60% power setting.

15.0 METHOD PERFORMANCE

15.1 Refer to EMT SOP # 109, 111, 118 and 119, analysis by ICP-MS and ICP-AES, for method performance.

15.2 *Method performance is detailed in the EMT Quality Assurance Manual, which includes sections on Method Startup, Reporting Limits, Method Detection Limits (MDLs), Method Control, and Initial Demonstrations of Capability (IDC).*

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 Clean up acid spills by neutralizing the spill and disposing of it with the materials in the spill kits.

16.2 Dispose of all acid wastes (samples, expired standards, rinse solutions, etc) in the Nitric acid (HNO_3) waste containers on the counter in the rear of the 8100 N. Building. A licensed waste hauler picks up the collected waste.

16.3 Old standard bottles to be disposed of (after solution poured off into the acid waste container) should be generously rinsed three times with water before being discarded. Many of the polypropylene containers can be recycled after rinsing.

16.4 *Refer to EMT "Quality Manual" for samples disposal used in this SOP.*

17.0 TABLES, DIAGRAMS, FLOW CHARTS, INSTRUMENT MAINTENANCE

17.1 Refer to Appendix A for the Spike preparations and standards holding times.

17.2 Refer to Appendix B for Calibration of MPS 1000.

17.3 Refer to Appendix C for Microwave power measurement for Calibration of MARS 5 microwave.

17.4 *Refer to Appendix D for MARS5-Temperature Calibration- RTP-300 Plus.*

17.5 *Refer to Appendix E for Microwave operating power setting.*

17.6 Refer to Table 1 for Event Sequence/Procedure.

17.7 Fume hood needs to be cleaned and vacuumed daily to ensure proper function and to prevent contamination.

17.8 Area must be kept clean and organized.

18.0 REFERENCES

18.1 CEM Operation Manual, Model MARS 5

18.2 CEM Operation Manual, Model MSP 1000

18.3 *Laboratory Quality Assurance Manual, Environmental Monitoring & Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.*

18.4 SW-846, Test Methods for Evaluating Solid Waste

Appendix A

ICP and ICP-MS Spike Solution Preparation for Methods 3015, 3050, and 3051 (Including E-Products)

Spikes for the ICP and ICP-MS are labeled as TM-## for total metals spike, O-## for the odd spike (minerals) and Gold, and L-## for Lucent spike. The ## refers to a sequential numbering system started from 01 and one digit is added each time the spike is re-made to give each solution a unique lot number. As example, a current solution of odd spike (O-44) when it is re-made the new solution will be O-45. All solutions of spikes have a shelf life (expiration date) of three months at which time they need to be re-made. Each solution, elements contained, and preparation sequence are outlined below for each individual solution. All solutions to be put into reagent books and labeled according to SOP for each prep method.

TM (Total Metals) Spike Preparation:

Compound/Standard (from individual stock solutions):	Initial conc. (mg/L):	mL used:	Total volume (mL):	Final Conc. (mg/L):	Spiked sample value (mg/L):
As,Ba,Cr,Cu,Mn,Ni,Pb,Se,Sb,Ti,Tl, & Zn	1000	10		50	1.0
Fe & Sn	10000	1		50	1.0
Ag, Be, & Cd	1000	1	200	5	0.1
B	1000	5		25	0.5
P	10000	5		250	5.0
HNO3	Conc.	20		10 %	NA

(Use 1mL of spike per 50mL of sample)

O (Odd Metals) Spike Preparation:

Compound/Standard (from individual stock solutions):	Initial conc. (mg/L):	mL used:	Total volume (mL):	Final Conc. (mg/L):	Spiked sample value (mg/L):
Al, Co, Mo, & V	1000	4		20	1.0
Ca, Mg, K, & Na	10000	2	200	100	5.0
HNO3	Conc.	20		10 %	NA

(Use 2.5mL of spike per 50mL of sample)

O (Odd Metals for gold) Spike Preparation:

Compound/Standard (from individual stock solutions):	Initial conc. (mg/L):	mL used:	Total volume (mL):	Final Conc. (mg/L):	Spiked sample value (mg/L):
Au	1000	5	100	50	1.0
HNO3	Conc.	10		10 %	NA

(Use 1mL of spike per 50mL of sample)

L (Lucent Metals) Spike Preparation:

Compound/Standard (from individual stock solutions):	Initial conc. (mg/L):	mL used:	Total volume (mL):	Final Conc. (mg/L):	Spiked sample value (mg/L):
Bi, Pt, Sr, & Ta	1000	5		100	1.0
Pd and Te	10000	0.5	50	100	1.0
HNO3	Conc.	5		10 %	NA

(Use 1mL of spike per 100mL of sample)

Appendix B

Calibration of MPS 1000

1.1 Measure the power at 20%, 40%, 60% power setting

1.2 Weigh 1,000.0 g+/-0.1g of reagent water into a 1L Teflon beaker and equilibrate to room temperature (23+/-2 °C). Measure temperature to +/-0.05 °C.

1.3 Place the covered beaker in the microwave oven for 2 minutes at the desired partial power setting. The unit's exhaust should operate normally.

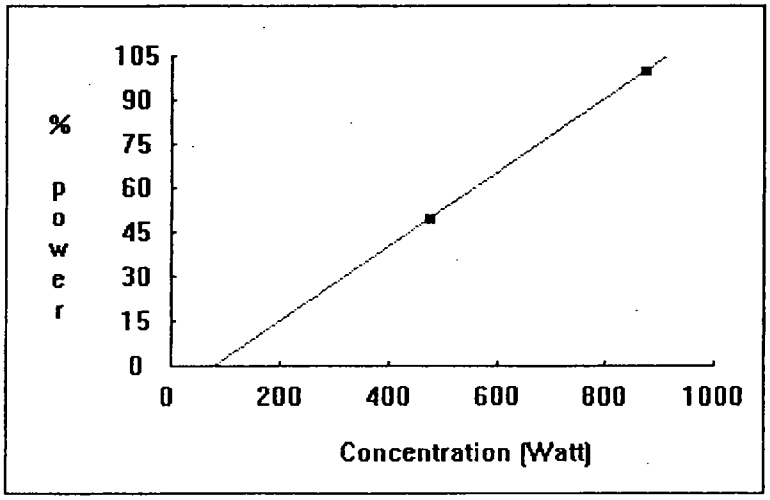
1.4 After 2 min remove the beaker from microwave, immediately insert stirring bar and stir vigorously on the stirring plate.

1.5 Within the first 30 seconds measure and record the temperature to +/-0.05 °C. (A new sample should be used for each measurement. If the water is reused both the water and the beaker must have returned to 23+/-2 °C).

1.6 Calculate absorbed power for each power setting:

Power (Watt) = **(Change in Temperature [°C]) X (34.86)**

1.7 Construct 2-point line by plotting power in Watts versus 20% and 60% power setting. Using 2-point line determine the power "watt" corresponding to 40% power setting. The measured power "watt" at 40% setting should not differ more than 10 W from the value obtained from the line.



1.8 If the difference is more than 10 W, perform the multiple point calibration using points clustered around the working power ranges in 10% power increments to determine non-linear deviation in any region.

1.9 Check power "watt " at 40% for MPS 1000 quarterly. If the measured absorbed power does not correspond to the specified value from the calibration within +/-10 W, the entire calibration should be reevaluated.

Appendix C

Microwave power measurement for Calibration of MARS 5 microwave

1.1 *Install the turntable in the microwave cavity.*

1.2 *With the main menu displayed, highlight "Load method". Press the "SELECT" key. The "Directory Menu" screen will appear.*

1.3 *Use the arrow keys to highlight "CEM Directory". Press the "SELECT" key. The "CEM Menu" screen will appear.*

1.4 *Use the arrow keys to highlight "1200W Power Test". Press the "SELECT " key to return to the main menu.*

1.5 *Place 1000mL of ambient temperature (18-22 °C) de-ionized water in a 1000-mL Teflon beaker.*

1.6 *Using a thermometer with 0.1 C gradations, measure and record the initial water temperature, Ti. Ensure that the thermometer is immersed to its indicated immersion line prior to reading the temperature.*

1.7 *Remove the thermometer from the beaker. Carefully place the beaker in vessel #1 position on turntable.*

1.8 *Gently close the door to avoid spilling any of the water.*

1.9 *Press "Start".*

1.10 *At the end of the programmed time (2 min.), remove the beaker from the microwave cavity. Stir the water thoroughly for 30 seconds, then measure and record the peak temperature reading. This is the final temperature Tf. The microwave power output is calculated as follows:*

$$\text{Power in Watts} = 35(Tf - Ti)$$

1.11 *If the measured power is below 1020W, repeat the microwave power measurement. If the power remains less than 1020W, the instrument is not producing adequate microwave power at the 1200W selection.*

1.12 *Repeat steps 1.2 through 1.9 for the 600W power test.*

1.13 *If the measured power is below 510W, repeat the power measurement. If the power remains less than 510W, the instrument is not producing adequate microwave power at the 600W selection.*

1.14 *If the instrument is not producing sufficient wattage, refer to the Troubleshooting Guide manual.*

Appendix D

MARS5-Temperature Calibration- RTP-300 Plus

1.1 From the Main Menu, press the "SETUP" key.

1.2 Using the arrow keys, highlight "Select Sensor". Press the "SELECT" key.

1.3 Using the arrow keys, highlight "Temperature sensor". Press the "SELECT" key.

1.4 Using the arrow keys, highlight "RTP-300Plus". Press the "SELECT" key.

1.5 Using the arrow keys, highlight "Enter GF number". Press the "SELECT" key.

1.6 Use the numerical keys to enter the GF number printed on the RTP-300 Plus probe. Press the "SELECT" key to return to the calibration screen.

1.7 Using the arrow keys, highlight Calibrate RTP-300 Plus". Press the "SELECT" key.

1.8 Insert the RTP-300 Plus into the connector in the center of the instrument.

1.9 Place the RTP-300 Plus in a beaker of water. Using the numeric keyboard, enter the temperature of the water as indicated on the thermometer.

1.10 Press the "SELECT" key. The "current temperature" on the screen should read the same as the temperature entered in step 1.9.

1.11 If necessary, repeat steps 1.9 and 1.10 to verify proper calibration of the RTP-300 Plus.

Appendix E

Microwave Operating Power Settings:

References: CEM manuals for MSP 1000, and High-Throughput Pb application sheet.

MSP-1000 (950 W system, - 3°s W/ Vessel)

# of Vessels:	6	8	9	10	12	
Power Settings:	36%/26%	43%/29%	46%/30%	50%/32%	56%/34%	*Wastewaters (20 minutes)*
Power Setting:	33%	40%	43%	46%	53%	*Solids (10 minutes), Current*

MSP-1000 (950 W system) for 36 sample carousel (60 minutes)

# of Vessels:	8	12	16	18	20	24	28	32	36
Power Setting:	9%	13%	18%	20%	22%	27%	31%	36%	40%

Settings based on 1.51 times more wattage than MDS-2000 and MDS-81D in which studies were done (0.663% W of MDS-2000)

Table 1

Event Sequence / Procedure:

Sample pick-up, login, preservation, and labeling
|
Microwave Equipment calibrated
|
Vessels needed cleaned and dried
|
Set-up digestion batch, add all needed QC samples and spikes
|
Place all samples in carousel, balance if needed
|
Digest sample batch
|
Allow samples to cool
|
Enter all information to Omega and print labels
|
Pour samples into labeled containers, filter if needed
|
Give digestates to analysts for metals determination

TITLE: METHOD 200.7. DETERMINATION OF METALS AND TRACE ELEMENTS IN WATER AND WASTES BY INDUCTIVELY COUPLED *ARGON* PLASMA *OPTICAL* EMISSION SPECTROMETER (ICP-*OES)*

KEY WORDS: Not applicable.

COMMENTS: The SOP was revised to convert to a new format and to specify the sources of standards used for preparation of ICV (secondary source) and CCV (the source used for instrument calibration).
The installation of new ICP-OES was completed 9/20/07.
Italicized items indicate changes from the last revision.

REVISED BY: Mioara Bratian October 31, 2007

APPROVALS:

METAL GROUP LEADER:  October 31, 2007
Mioara Bratian

QUALITY ASSURANCE AUDITOR:  October 31, 2007
Mary Lubitov

METHOD 200.7. DETERMINATION OF METALS AND TRACE ELEMENTS IN WATER AND WASTES BY INDUCTIVELY COUPLED ARGON PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES)

1.0 SCOPE AND APPLICATION

1.1 The analysis by *ICP-OES* allows the determination of the target elements with high precision (uncertainty in the low percentage range) and/or high sensitivity.

1.2 Inductively coupled plasma-atomic emission spectroscopy *(ICP-OES)* determines trace metals in an aqueous solution. The method is applicable to all of the elements found in the table in Sec.17.1.

2.0 SUMMARY OF METHOD

2.1 Samples must be solubilized or digested using appropriate sample preparation methods (refer to the digestion method for the sample size).

2.2 The analysis in this method involves multi elemental determinations by *ICP-OES* using sequential or simultaneous instruments. The instrument spectrometers measure characteristic atomic-line emissions with photomultiplier tubes (PMT). Aqueous samples are nebulized with Argon gas and the resulting aerosol flows through the spray chamber to eliminate large droplets. After the chamber the aerosol is carried to the torch where it is dried, atomized, and ionized in the plasma that is produced through radio frequency coupling. The excited atoms release specific wavelengths of light as they lose energy gained in the plasma producing element specific emission spectra. The spectra of light emitted are dispersed by a grating after entering the optics through the entrance slit and the intensities of the line spectra are monitored at specific wavelengths by the PMT where photocurrents are processed, integrated, and put into concentrations (mg/L of atoms present) by a computer system.

2.3 Background correction is required to compensate for variable background contribution by interfering structure near the analyte of interests' wavelength. Background must be measured adjacent to the analyte wavelength during analysis where the least amount of background structure present. The background correction subtracts out the raised baseline found at the correction point chosen to eliminate false positive results. Inter-element corrections may also be used as background corrective measures when spectral overlaps develop from high concentrations of interfering elements.

2.4 Samples after preparation (digestion) become 50mL of acidified solution ready to be analyzed. 5 to 15mL of solutions are placed on the instruments' auto sampler and the sequence is programmed into the computer to set up the batch run.

3.0 DEFINITIONS

Refer to EMT "Quality Manual" for definitions of terms. No definitions unique for the test.

4.0 SAFETY

4.1 Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon tanks and fittings.

4.2 The ICP instrument is fully interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures and other hazards. At no time

should the operator attempt to disable these interlocks or operate the instrument if any safety interlock suspected to be disabled.

4.3 Always add the concentrated acid slowly to the water (not water to acid) to avoid a reaction.

4.4 *All samples handled should be considered hazardous and the appropriate PPE should be worn when running this test (safety glasses, gloves, and a lab coat).*

4.5 *Each chemical compound used in this method should be treated as a potential health hazard. MSDS for chemicals are kept at the front desk of 8100 N Austin building. Be aware of the hazardous effects of Hg and the reagents when inhaled or contacted with skin.*

4.6 *The use of laboratory equipment and chemicals exposes the analyst to several potential hazards and good laboratory technique and safety practices should be practiced at all times including the use of safety glasses, laboratory coats and acid resistant gloves when handling samples or reagents, or when in the vicinity of others handling these items.*

4.7 *Spilled samples and reagents should be cleaned up from laboratory surfaces immediately. Acidic and Alkaline spills should use spill kit "pigs" for caustic spills.*

4.8 *All additional company safety practices and procedures should be followed at all times.*

4.9 *Also refer to Chemical Hygiene plan in 8100 Conference room.*

NOTE: Please refer to the latest version of EMT' s Chemical Hygiene Plan (CHP) for more comprehensive and authoritative safety information. The information provided in this section is to be used as guidance. The information given in the CHP supercedes the information provided here.

5.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

5.1 Samples are to be collected in metal free containers such as glass quart jars or plastic pint jars with plastic or Teflon lids.

5.2 All glassware used in the lab for metals should be cleaned with NOCHROMIX and/or soaked in *10% HNO_3* and rinsed thoroughly with DI water before use.

5.3 To delay the chemical and biological changes that inevitably continue after the sample is removed from the parent source, a preservation technique is required. Metal ions may precipitate as hydroxides or form complexes with other constituents. Cations or anions may change valence states under certain reducing or oxidizing conditions while other constituents may dissolve or volatilize with the passage of time. Metal cations may also absorb onto surfaces of the container (glass, plastic, etc.), such as iron and lead. Preserve the liquid and extract samples for metals analysis to a pH < 2 with HNO_3, which keeps the metal ions in solution and delays the ionic exchange between the metallic ions and the surface of the container.

5.4 Preserve drinking water samples with HNO_3 to pH < 2 for at least 16 hours before the analysis. Check pH immediately prior to analysis to ensure that the samples were properly preserved. If for some reason the sample pH is verified to be >2, add more acid and hold the sample for 16 hours. Then verify the pH is < 2. Turbidity should also be measured for all drinking waters, since samples with turbidity of up to 3 NTU can appear clear to the unaided eye.

5.5 All samples should be preserved as soon as possible after collection and kept on ice if not preserved. After receipt, samples are to be logged in and stored in the login cooler at 4 °C.

5.6 Even though the holding time for metals is 6 months, it is best to analyze the samples as soon as possible after collection or preparation.

6.0 INTERFERENCE

6.1 Spectral Interferences - caused by overlap of a spectral line from another element, or unresolved overlap of molecular band spectra, background emission from continuous or recombination phenomena stray light from the line emission of high concentration elements.

6.1.1 Spectral overlap may be avoided by using alternative wavelength or computer-corrected by measuring the interfering elements and use IEC's (inter-element correction factors).

6.1.2 Unresolved overlap (OH on both Be and V) requires selection of an alternate wavelength or analysis on ICP-MS.

6.1.3 Background contribution and stray light from high-concentration elements (many samples with high levels of various elements showing up across spectra) can usually be compensated by a background correction adjacent to the analyte line.

6.2 Physical Interferences

6.2.1 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. Diluting the sample that minimizes the matrix interferences can reduce the physical interferences.

6.2.2 Samples with high dissolved solids can cause salt buildup at the tip of the nebulizer or torch injector tip which affects aerosol flow rate and may cause instrumental drift. Diluting the sample, changing the nebulizer, and removing salt buildup by cleaning the torch and the injector tube could reduce the problem. Adjustments on argon flow and pump rate could improve the instrument performance, it is generally only a short-term fix until the system is thoroughly cleaned.

6.3 Chemical Interferences

6.3.1 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects.

6.3.2 Chemical interferences are highly dependent on matrix type and the specific analyte element. Chemical interferences can be minimized by matrix matching, and by standard addition procedures.

6.4 Memory Interferences

6.4.1 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition in the uptake system and from the buildup of sample material in the plasma torch.

6.4.2 Flushing the system with a rinse blank between samples can minimize memory effects. A rinse period of at least 60 sec. between sample and standards is required. Continued memory problems warrant a cleaning of the sample uptake system, possibly including the torch if a build-up of material is observed.

6.4.3 Samples with an analyte reading of greater than 20 mg/L may cause a memory effect (Ag, Mo, Ba, and Na seem to get "stuck" often, requiring long rinse-outs and baseline checks prior to continuing the analysis)

7.0 EQUIPMENT AND SUPPLIES

7.1 Argon gas supply: Industrial argon, grade 5, 99.998% purity.

7.2 Auto pipettes: In ranges from 1 to 10mL, to be calibrated monthly to a tolerance of 0.6% at fixed settings on the 5 and 10mL pipettes.

7.3 Electronic Analytical balance "Sartorius AC-210S", capable of weighing to the nearest 0.0001g. The balance should be calibrated daily when in use.

7.4 Inductively coupled argon plasma *optical* emission spectrometer (*ICP-OES*)-computer controlled with background correction and IEC capability-*ICAP-6300*, computer, printer, and auto sampler.

8.0 REAGENTS AND STANDARDS

8.1 ***Reagents and Standards Labeling Requirements.***

8.1.1 *Container of commercially bought standard and reagent must have label with Standard/Reagent Name, Initial Concentration, Manufacturer, Lot Number, Expiration Date, Date Received, and Date Opened.*

8.1.2 *Container of prepared solution must have label with Name of Solution, Concentration, Expiration Date and Traceability to the preparation.*

8.1.3 *Reagents and Standards Log Book must have the following information: Standard/Reagent Name, Manufacturer, Lot Number, Date Opened, Expiration Date, Initial Concentration, Initial Volume, Final Volume, Final Concentration, Initials of Person received and prepared the solution.*

NOTE: *Follow the expiration date listed on the stock reagent or listed below by prepared reagent. Re-order reagents and stock solutions as needed.*

NOTE: Low trace metals grade reagents and certified standards shall be used in the test

8.2 Blanks: Two types of blanks are required for the analysis of samples. The calibration blank is used in establishing the analytical curve and the method blank is used to identify possible contamination resulting from either the reagents (acids) or the equipment used during sample processing including filtration.

8.2.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations.

8.2.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

8.3 Certified individual and custom blended standard stock solutions traceable to NIST for all analytes of interest.

8.4 DI water, ASTM type II.

8.5 Nitric acid (concentrated), HNO_3

8.6 Standards Preparation. See tables in Sec.17.2 and 17.3. Standards need to be re-made according to the following schedule: <1 mg/L - monthly. 1-20 mg/L – every 3 months. 20-300 mg/L- every 6 months. 300-1000 mg/L standards - every 12 months.

8.7 The IEC standards 100 mg/L are prepared by adding 1mL of 1000 mg/L interfering element solution to a 100-mL flask with DI water and 10 mL of HNO_3 conc., and diluting to mark. Be careful not to include more than one interfering element for one analyte of interest in each IEC solution. Multiple IEC solutions might be needed for multiple analytes of interest.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Calibrate instrument daily starting with calibration blank (Standard#1), proceed through 4 multi-element standards (refer to Sec.17.2). The calibration equation formula is in Sec.11.1.

9.2 The correlation coefficient for the calibration needs to be > 0.995 for all elements. If the coefficient is lower for any needed element, the instrument needs to be re-calibrated for any needed elements included in the run before analysis can begin. If calibration fails again, problem needs to be investigated and corrected. There could be contamination in the blank, one of the standards may be diluted wrong, or the instrument uptake system may not be functioning properly (the last two should effect all elements, a contamination or memory effect may only effect one element)

9.3 The Initial calibration verification, ICB and ICV should be analyzed immediately following the initial calibration. To be acceptable an ICB should be <IDL (as +/-3 SD of calibration blank). *The ICV is prepared from an independent (secondary source) material at mid-range of the calibration curve and* the recovery of the ICV must be within 95 -105% and the %RSD of the 3 replicates needs to be < 3%.

9.4 The continuous calibration verification, CCV, should be analyzed following the initial calibration verification, at the end of the run and after every tenth injection/sample. *The CCV is made from the same material as the calibration standards at mid-range of the calibration curve*; the recovery must be within 95 -105%.

9.5 The continuous calibration blank, CCB, should be analyzed after every tenth injection/sample, and at the end of the sample run. To be acceptable the CCB should be <IDL (as +/-2 SD of calibration blank). If out of range analyze 2 more times and average all 3 results. The average of 3 should be within the limits.

9.6 Low Level check standard (PEM = Performance evaluation mix) should be analyzed at the beginning of the analytical run. PEM recovery must be within 80 -120%.

9.7 If the calibration cannot be verified within the specified limits, reanalyze failed QC sample. If the second analysis fails, the analysis must be discontinued and the cause of failure determined and corrected. The instrument should be recalibrated. All samples following the last acceptable CCV or CCB and prior to a failed CCV or CCB must be reanalyzed. Acceptable calibration verification standards and blanks for all analytes of interest must bracket samples. The results can be reported only if the CCV and CCB fail high and the failed analytes are not detected in samples. Failure of the ICV or CCV generally is due to a bad calibration or instrumental drift. A CCV after the samples with high viscosities may be suppressed, which can be re-checked once after allowing for a rinse-out. If passing on the re-check, prior samples should be run with dilution or checked with a bench spike for matrix effects.

10.0 PROCEDURE

10.1 Background Correction Setting

10.1.1 To set the background correction point, analyst(s) will need to scan numerous sample types and matrices that will normally be run as well as mixed interferent standard solutions for all wavelengths that will be run.

10.1.2 The various scans should be overlaid with a blank solution (baseline solution) to find where the interferent peaks or raised backgrounds will occur near the wavelength of interest. The background point should be selected to the left or right of the analytical zero where no interfering structure occurs.

10.1.3 The background point must also lie far enough away from the analytical zero (exact analyte wavelength) so that a high sample concentration with a large peak will not "self-subtract" itself if the analytes' peak itself hits the background point.

10.1.4 Two background points are not recommended except in special cases to eliminate a sloping background, as dual points will decrease sensitivity.

10.2 Turn on hoods and chiller systems.

10.3 Bring up the computer software.

10.4 Start the instrument by igniting the plasma (also refer to manufacturer's instrument manuals for instrument specific instructions)

10.5 Set instrument to operating conditions (nebulizer pressure, pump rate, Argon flows, etc.) needed for the day's analysis, method run, and let warm-up (generally a half an hour is sufficient)

10.6 Perform any needed diagnostic stability tests (refer to manuals) and align spectrometer

10.7 Proceed to type in samples to be run in Auto sampler table, pour out samples and add into appropriate positions in auto sampler rack.

10.8 Proceed with samples analysis.

10.8.1 Wastewater Total Metals

10.8.1.1 Unlike the drinking water and dissolved samples (see below), the digested wastewater samples can be put directly onto the instrument and run after sample prep is complete.

10.8.1.2 Refer to digestion methods and SOP's regarding the prep of wastewaters for digestion (Method 3015, SOP #101).

10.8.2 Drinking Water

NOTE: Drinking waters run without digestion are to be run with 1% HNO_3 standards and blanks, all other analysis uses 9% HNO_3.

10.8.2.1 To determine whether digestion of the sample is required, the turbidity of the acidified sample must be measured using an approved method and only after preservation is complete (SOP #064). Preservation is complete after the acidified sample has been held for 16 hours. Before sample processing is started, sample pH must be verified to be less than 2. If pH greater than 2, acidify sample to < 2 and wait 16 hours to do turbidity

screen. Document the pH of the sample in the turbidity book (refer to SOP #064 and Turbidity Methods).

10.8.2.2 For the "direct analysis" of total recoverable analytes in drinking water samples containing turbidity <1 NTU, add an appropriate volume of (1+1) HNO_3 to an unfiltered acid preserved sample aliquot to adjust the acid concentration of the aliquot to approximate a 1% (v/v) HNO_3 solution (add 1mL concentrated HNO_3 to 99mL of sample). An allowance for sample dilution should be made in the calculations.

10.8.2.3 For the determination of total recoverable analytes in drinking water samples with >1 NTU turbidity, transfer 100mL aliquot from a well mixed, acid preserved sample to a 250mL Griffin beaker (smaller sample aliquot volumes may be used when necessary). Set up additional beakers for blank, fortified blank, sample spike and spike duplicate.

10.8.2.4 Add 2mL (1+1) HNO_3 and 1.0mL of (1+1) HCl to the beaker.

10.8.2.5 Place the beaker on the hot plate for solution evaporation.

10.8.2.6 Reduce the volume of the sample aliquot to about 20mL by gentle heating at 85 °C uncovered and 95 °C covered with a watch glass.

10.8.2.7 Reflux the sample for additional 30 minutes on the hot plate and allow the beaker to cool.

10.8.2.8 Quantitatively transfer the sample solution to a graduate cylinder and bring the volume to 50mL with DI water (bring to 25mL of the volume if low level needs to be achieved).

10.8.2.9 Filter the sample if suspended solid is present.

10.8.3 Dissolved Metals

10.8.3.1 For the determination of dissolved analytes in ground and surface waters, unpreserved sample should be filtered through 0.45um membrane filter. Acid preserves the samples to match the matrix of the calibration standard only after the filtration. Add 5mL of concentrated HNO_3 to 45mL of sample to matrix match and analyze. Allowance for sample dilution should be made in the calculation.

10.8.3.2 To reduce potential interferences, dissolved solids should be <0.2% (w/v).

10.8.3.3 Dilute the samples to reduce the physical interferences.

10.8.3.4 Salt buildup at the tip of the nebulizer due to high dissolved solids affects aerosol flow rate and causes instrumental drift. This could also be controlled by diluting the samples high in salts and dissolved solids.

10.8.3.5 If a precipitate is formed during acidification, transport, or storage, the sample aliquot must be digested according to Drinking Water digestion method.

10.8.3.6 Use Drinking Water digestion method if low level needs to be achieved.

10.9 Shutdown

10.9.1 After sample run complete and final QC checks are verified to be acceptable, allow instrument to flush out any sample residue (turning up pump rate and nebulizer gas flow also aids in cleaning system).

10.9.2 By alternating between rinses such as 9% HNO_3, DI water and matrix blanks most residues should be removed. Allow longer time if samples with high levels of analytes were run.

10.9.3 Shutdown and turn off instrument according to instrument manual (usually left in "stand- by" mode).

10.9.4 Preventive maintenance should include: minimum weekly check of pump tubings, inspection of torch & injection tube, and nebulizer changing.

10.9.5 Document all the actions in the maintenance book. More frequent maintenance should be done if necessary. Refer to manufacturer's manual book.

10.10 Appendix A contains "Event/Sequence Procedure".

11.0 CALCULATIONS

11.1 Calibration equation formula:

Concentration = **(slope) (Intensity) + Y Intercept.**

11.2 The final result is calculated as follows:

$$\text{Final concentration of analyte (mg/L)} = \frac{\mathbf{A \times B \times C}}{\mathbf{mL\ of\ sample\ digested}}$$

Where:

A = Instrument reading (mg/L)
B = Final volume of digested sample (if digested) in mL.
C = any secondary dilution factor after digestion

11.3 *Calculation of the common quality control parameters (e.g., percent recovery, percent difference, relative percent difference (RPD), and relative standard deviation (RSD)), are given in the EMT Laboratory Quality Assurance Manual.*

12.0 QUALITY CONTROL

12.1 **Duplicate sample (DUP)** must be analyzed at a frequency of one per batch. Duplicates must have a Relative Percent Difference (RPD) of < 20%.

12.2 **Control charts** are to be generated for LCS, MS/MSD % recovery and RPD.

12.2.1 The limits generated in the laboratory should be used for the data evaluation if they are more stringent than the pre-established limits. The control limits should be updated semiannually. Control charts should be reviewed monthly for systematic trends.

12.2.2 The control charts are put into a tabulated spreadsheet after creation from the LIMS system and stored also on the "G" drive in the "&Exceldoc" folder in the subfolder "control limits" for easy reproduction and analyst review.

12.3 **IEC K_1 Coefficient calculation Verification:** Every six months the IEC K_1 coefficients need to be verified and corrected if needed. To verify the IEC corrections individual 100 mg/L solutions of known interfering elements are analyzed as samples with the current IEC corrections turned off. All negative or positive results greater than MDL for any element is suspect of needing an IEC. The suspect interferences should coordinate with the initial IEC set up and the continuing IEC check standards. Each suspect result is the interference caused by the 100 mg/L element on a

given analyte. These values need to be divided by 100 and entered into the instrument as the K_1 correction value. Both positive and negative results are to be used and entered as positive or negative in the K_1 value. In this fashion, all IEC's and K_1 values are refreshed, verified, and checked every six months.

12.3.1 For example, upon running 100 mg/L solutions of Cu and Ni as "unknowns" the following occurred: On the 100 mg/L Cu solution Ag had a reading of - 0.082 and Cd had a reading of 0.040 mg/L

12.3.2 On the 100 mg/L Ni solution Ag had a reading of 0.065 mg/L.

12.3.3 Both readings for Ag and Cd are greater than MDL, so IEC is suspect. The K_1 values needed are: -.00082 for Cu and .00065 for Ni interferences on the Ag analyte and 0.0004 for Cu interference on the Cd analyte.

12.3.4 After all updates are loaded into the instrument, the appropriate IEC check solutions need to be made and the checked to verify the corrections prior to analysis for the day.

12.4 **Inter Element Correction (IEC) solution** is analyzed daily at the beginning of the first analytical run to verify the inter element interference (K_1) correction factors. 100 mg/L interfering analytes should have a recovery within 90-110% to correctly calculate IEC factors. The measured concentration from interfering analytes should be within +/- 2 X MDL. If outside this range, the correction factor needs to be modified and the analyte re-run to ensure new correction is appropriate. Samples run with the wrong correction factor will calculate erroneous results for the sample when the interfering element is present in a high enough concentration to cause a significant effect to the raw value. Changes for correction factors need to be put on cover sheet included with run data.

12.5 **Laboratory Control Sample (LCS)** should be analyzed with each batch of 20 samples or fewer. The recovery of any analyte of interest must be within 85-115% or within the established control limits (whichever is more stringent). Batches with a LCS outside limits will need to be re-digested and re-analyzed. Failed LCS could be a prep problem or indicative of instrument drift. The problem needs to be investigated and documented.

12.6 **Linear dynamic range (LDR)** must be established for all elements. A minimum of 6 evenly spaced standards in the concentration range from 0 to 500 mg/L are run to see when the linearity fails, which is when the standard recovery drops below 10% of the target value. The standard prior to the failed standard is the upper limit of the LDR. If a more precise value is needed, another curve with more closely spaced concentrations around the initial LDR may be run to narrow the result down. A low level standard near PQL needs to be included with the LDR study to prove linearity at the low end of the calibration. The LDR needs to be verified every six months or whenever a change in the instrument performance observed. The samples containing analytes of interest or interfering elements above LDR need to be diluted.

12.7 **Matrix Spike** is analyzed at frequency of one MS per 20 samples. Acceptance recovery range for MS/MSD is 70-130%. Acceptable RPD limit is < 20%. Use in house generated limits if they are more stringent than the preset limits.

12.8 **Method Blank (MBLK)** should be analyzed one per batch of 20 samples or fewer. When MBLK values exceed 10% or more of the analyte level determined for a sample or 2.2 of the MDL for that analyte (whichever is less), the samples needing the failed analyte should be re-digested and reanalyzed unless the samples needed are non-detect for that analyte. A failed blank may be indicative of a baseline drift or contamination in the sample prep. Continuation of a problem will need to be investigated and documented.

12.9 Samples out of range need to be checked for matrix, memory, or background interferences through the use of Dilution Test or MSA.

12.10 Dilution test can be applied if the concentration of analytes in the solution at least x 10 times the MDL level. The results from different dilutions should agree within 10%. RPD > 10% indicates matrix specific problem.

12.11 To verify matrix interference perform bench spike. The level of spike needs to be at least 20 x MDL or 50-150% of sample background, whatever is higher. The ratio spike volume to sample volume should not exceed 1:10. The recovery of post-digest spike outside of 85-115% indicates matrix effect. The samples with matrix specific problems need to be flagged when reported.

12.12 Failure of all metals in MS might be a sign of a digestion error or failure to spike sample. Re-digest the sample with the failed MS if the problem is not matrix related. If the sample does not appear to be inhomogeneous, the problems might be the uptake system. The problem needs to be located, addressed and documented. If the second digestion fails, the data may be reported with a note explaining the nature of the problem.

12.13 *For Quality Control Samples with Limits and Frequencies see Sec 17.4*

13.0 REPORTING

For reporting refer to the most current revision of SOP # 209.

14.0 DEVIATIONS FROM THE REFERENCE METHOD

14.1 Samples with non-detect analytes and associated with ICB/CCB < 2.2 x MDL can be reported.

14.2 MDL may be based on IDL of 200.7, 6010B, or MDL. Whichever is highest is used for MDL and PQL determination to ensure value is not unreasonably low.

14.3 Standard solutions are prepared in 9% HNO3 to match the samples matrix after the digestion.

14.4 Requirement to analyze low level check standard is incorporated to comply with NELAC standard.

14.5 Analytes above calibration range but within 90% of Linear Dynamic Range will be reported with flag.

14.6 If CCV fails in the first attempt re-run CCV one more times. If the re-runs of CCV fail low, correct the problem, document it, re-calibrate and prior sample section needs to be re-run for failed analytes. If initial check or re-runs of CCV fails high, may report elements in the section as long as they are non-detected or less than reporting limit.

15.0 METHOD PERFORMANCE

15.1 All files for the IDC's, MDL's, LDR's, and IDL's are tabulated and stored on the network drive directory of "G" in the file "&Exceldoc" under each of their representative instrument and file type headings.

15.2 **The Instrument Detection Limit (IDL)** is determined by running calibration blank 7 to 10 times. The IDL is calculated as standard deviation of replicates multiplied by 3.

15.3 **Demonstration of Capability (DOC)** should be performed for all analytes of interest prior to analyses conducted by this method. DOC need to be repeated every time there is a change in samples preparation, instrumentation, or personnel. Four standards at a concentration ~ 10 times MDL should be prepared and analyzed as regular samples. The source of the spiking solution

should be unassociated with the source of calibration standards. The % Recovery for each replicate must be within 80-120% and the RSD % must be 20% or less for the study to be acceptable.

15.4 **Method detection limit (MDL)** study should be performed annually for all analytes of interest. The matrix used for MDL study should contain analytes at level 2-4 times of anticipated MDL. The study is performed following SOP # 218 "MDL study guidance". Reporting limit is established as ~ 3 times the MDL. If calculated MDL is lower than the IDL, the IDL value is considered the MDL.

15.5 *Method performance is detailed in the EMT Quality Assurance Manual, which includes sections on Method Startup, Reporting Limits, Method Detection Limits (MDLs), Method Control, and Initial Demonstrations of Capability (IDC).*

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 Dispose of all acid wastes (samples, expired standards, rinse solutions, etc.) in the HNO_3 waste acid container located on the counter at the rear entrance of 8100 N. Austin. A specialized waste hauler then picks up the waste.

16.2 Clean up all spills of acid properly by neutralizing the spill and disposing of it.

16.3 Old standards' containers should be rinsed generously 3 times with tap water before disposing. Many of the polypropylene containers can be recycled after rinsing.

16.4 *Refer to EMT "Quality Manual" for samples disposal used in this SOP.*

17.0 TABLES, DIAGRAMS, FLOW CHARTS, INSTRUMENT MAINTENANCE

17.1 Elements

Element		Chemical Abstract Services Registry Numbers (CASRN)
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-39-3
Bismuth	*(Bi)*	*7440-69-9*
Boron	*(B)*	*7440-42-8*
Calcium	(Ca)	7440-70-2
Cadmium	(Cd)	7440-43-9
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Gold	*(Au)*	
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Manganese	(Mn)	7439-96-5
Magnesium	(Mg)	7439-95-4
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Phosphorous	*(P)*	*7723-14-0*
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2

Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Strontium	*(Sr)*	*7440-24-6*
Tantalum	*(Ta)*	
Tin	*(Sn)*	*7440-31-5*
Titanium	*(Ti)*	*7440-32-6*
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

17.2 Initial Calibration of Standards

Std	*Compound/Standard:*	*Initial conc. mg/L*	*mL used:*	*Total volume:*	*Final conc. mg/L*	*Holding Time*
	Custom Blend Std 1: Al, Sb, Cr, Au, Mo, Pd, Pt, Na, Sn, Ti, As, Co, Cu, Mg, Mn, Ni	*1000*	*10*		*10*	
#5	*Custom Blend Std 2: Ba, Be, B, Cd, Ca, Fe, Pb, Li, P, K, Se, Ag, Sr, Tl, V, Zn*	*1000*	*10*	*1 liter*	*10*	
	Be	*100*			*1*	*3 Months*
	Fe	*2000*			*20*	
	HNO_3	*conc.*	*90*		*9%*	
	Custom Blend Std 1: Al, Sb, Cr, Au, Mo, Pd, Pt, Na, Sn, Ti, As, Co, Cu, Mg, Mn, Ni	*1000*	*5*		*5*	
#4	*Custom Blend Std 2: Ba, Be, B, Cd, Ca, Fe, Pb, Li, P, K, Se, Ag, Sr, Tl, V, Zn*	*1000*	*5*	*1 liter*	*5*	
And CCV	*Be*	*100*			*0.5*	*1 Month*
	Fe	*2000*			*10*	
	HNO_3	*conc.*	*90*		*9%*	
	Custom Blend Std 1: Al, Sb, Cr, Au, Mo, Pd, Pt, Na, Sn, Ti, As, Co, Cu, Mg, Mn, Ni	*1000*	*2.5*		*2.5*	
#3	*Custom Blend Std 2: Ba, Be, B, Cd, Ca, Fe, Pb, Li, P, K, Se, Ag, Sr, Tl, V, Zn*	*1000*	*2.5*	*1 liter*	*2.5*	
	Be	*100*			*0.25*	*1 Month*
	Fe	*2000*			*5*	
	HNO_3	*conc.*	*90*		*9%*	
	Custom Blend Std 1: Al, Sb, Cr, Au, Mo, Pd, Pt, Na, Sn, Ti, As, Co, Cu, Mg, Mn, Ni	*1000*	*1.25*		*1.25*	
#2	*Custom Blend Std 2: Ba, Be, B, Cd, Ca, Fe, Pb, Li, P, K,*	*1000*	*1.25*	*1 liter*	*1.25*	

	Se, Ag, Sr, Tl, V, Zn					
	Be	*100*			*0.125*	*1 Month*
	Fe	*2000*			*2.5*	
	HNO_3	*conc.*	*90*		*9%*	
ICV (prepared from a source unassociated with calibration standards)						
Compound/Standard:		*Initial conc. (mg/L):*	*mL used:*	*Total volume:*	*Final Conc. (mg/L):*	*Holding Time*
Custom Blend Std 1: Al, Sb, Cr, Au, Mo, Pd, Pt, Na, Sn, Ti, As, Co, Cu, Mg, Mn, Ni		*1000*	*5*		*5*	
Custom Blend Std 2: Ba, Be, B, Cd, Ca, Fe, Pb, Li, P, K, Se, Ag, Sr, Tl, V, Zn		*1000*	*5*		*5*	
Be		*100*		*1 liter*	*0.5*	*3 Months*
Fe		*2000*			*10*	
HNO_3		*conc.*	*90*		*9%*	

17.3 *Low Level Check Standard*

Compound/Standard:	*Initial conc. mg/L*	*mL used*	*mL total*	*Final conc. mg/L*	*Holding Time*
CCV Solution: Al, Sb, Cr, Au, Mo, Pd, Pt, Na, Sn, Ti, As, Co, Cu, Mg, Mn, Ni	*5*	*0.5*	*100*	*0.05*	
Ba, Be, B, Cd, Ca, Fe, Pb, Li, P, K, Se, Ag, Sr, Tl, V, Zn *Be* *Fe*	*5* *0.5* *10*			*0.05* *0.005* *0.1*	*1 Month*
HNO_3 conc.	*conc.*	*9*		*9%*	

17.4 Quality Control Samples with Limits and Frequencies

QC Type	Waters: Drinkwaters
Method	200.7
IEC	At start of day </= to 2 X MDL
ICV	At start of every run within 5% of TV
ICB	At start of every run < 2 x STD Dev. **
*CCV	Every 10 injections within 10% of TV
*CCB	Every 10 injections < 2 x STD Dev. **
MBLK	Every digestion batch </= MDL, in 2.2 X MDL, < 10% sample
LCS	Every digestion batch within 15% of TV
MS	Every digestion batch within 30% of TV
MSD	Every digestion batch within 30% of TV, 20% RPD
PDS	As needed in 10% of TV, or interference present

QC Type	SW-846 (TCLP, Groundwater, solids)
Method	6010B
IEC	At start of day </= to 2 X MDL
ICV	At start of every run within 10% of TV
ICB	At start of every run </= to MDL or 3 x IDL
*CCV	Every 10 injections within 10% of TV
*CCB	Every 10 injections </= to MDL or 3 x IDL
MBLK	Every digestion batch of 20 </= MDL, < 5% Regulatory limit, < 10% sample
LCS	Every digestion batch of 20 within 20% of TV
MS	Every digestion batch of 20 within 25% of TV
MSD	Every digestion batch of 20 within 25% of TV, 20% RPD
PDS	As needed in 15% of TV, or interference present

** If ICB or CCB fails, may rerun. If 2 nd check fails, run a third and average. If average fails, correct problem and re-run samples.

*The results can be reported only if the CCV and CCB fail high and the failed analytes are not detected in samples.

18.0 REFERENCES

18.1 40 Code of Federal regulations, Appendix C to part 136, Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes. Method 200.7

18.2 *ICAP 6000 Series ICP-OES Spectrometer. Operator Manual.*

18.3 *Laboratory Quality Assurance Manual, Environmental Monitoring & Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.*

18.4 Method 200.7, Determination of metals and trace elements in water and wastes by ICP-AES. Revision 4.4 May 1994. US EPA.

18.5 Technical Notes on Drinking Water Methods, October 1994 -Section IV, Mandatory Method Modifications

APPENDIX A

Event Sequence / Procedure

Sample digestion by method 3015
|
Set up all samples after prep on Auto sampler and type into computer for run
(Also put into run log book)
|
Do any required maintenance prior to start of calibration
|
Ensure all standards to be used not expired
|
Calibrate instrument after warm-up
|
Make sure calibration and initial checks in acceptance ranges
|
Verify IEC coefficients acceptable at start of day / new analysis
|
Proceed to run samples and associated QC on run (CCB's, CCV's, etc.)
(ensure each digestion batch QC acceptable for all needed analytes)
|
Ensure final QC checks at end in acceptance range
|
Report results into computer (including any needed flags (higher limit due to dilution, MSA, etc.)
Shutdown instrument according to specific manufacturer

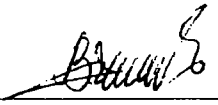
TITLE: DETERMINATION OF TRACE ELEMENTS IN WATER AND WASTES BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS), METHOD 200.8

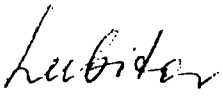
KEY WORDS: Not applicable.

COMMENTS: The SOP was revised to convert to a new format and to specify the sources of standards used for preparation of ICV (secondary source) and CCV (the source used for instrument calibration). Also Refer to Annex 3 and 4 for Calibration Standards and Instruments Check Standards Preparation Guide.
Italicized items indicate changes from the last revision.

REVISED BY: Mioara Bratian August 10, 2007

APPROVALS:

SAMPLE PREP. AND METALS GROUP LEADER:  August 10, 2007
Mioara Bratian

QUALITY ASSURANCE AUDITOR:  August 10, 2007
Mary Lubitov

DETERMINATION OF TRACE ELEMENTS IN WATER AND WASTES BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS), METHOD 200.8

1.0 SCOPE AND APPLICATION

1.1 ICP-MS determines trace metals down to sub ug/L levels in solutions derived from specified digestion procedures. The method determines dissolved, suspended, or total trace elements in wastewaters, drink waters, ground waters, surface waters, domestic and industrial wastes. Applied to all of the following elements found in the table in Sec.17.1.

1.2 ICP-MS has been applied to over 60 elements with detection levels typically below 0.02 ug/L. Use of an internal standard must be used when running by ICP-MS for metals determination.

1.3 With the exception of the silver, drinking water samples may be analyzed directly with a 1% nitric acid preserved sample without acid digestion provided the sample is properly preserved with nitric acid to a pH of < 2 and the turbidity is < 1 NTU. Silver is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, samples without acid digestion may have low recoveries of silver and for this reason it is recommended that samples be digested prior to the determination of silver (regardless of Turbidity). Digestion should be done to all samples excluding drinking waters and dissolved metals samples where total concentration is to be determined.

1.4 Dissolved elements need to be filtered through a 0.45-micron filter prior to addition of acid for preservation and matrix matching.

2.0 SUMMARY OF METHOD

2.1 Samples must be digested using appropriate sample preparation methods 3015, 3051 or 3050B for total metals analysis. Method 3005 is used as the filtration and preservation prep for dissolved metals. Refer to SOP #'s 101, 104, 106, and 117 respectively.

2.2 The analysis involves multi-elemental determinations by ICP-MS. The instrument measures ions created in radio frequency coupled plasma. Aqueous samples after digestion are nebulized with Argon gas and the resulting aerosol flows through the spray chamber to eliminate large droplets. After the spray chamber, the aerosol is carried to the torch where it is dried, atomized, and ionized in the plasma. The ions extracted out of the plasma pass through two cones via a vacuum interface into the mass spectrometer. The ions are sorted according to their mass to charge ratio by a quadrupole and quantified by the Electron multiplier (EM) mass detector using pulse detection for low amounts and analog signals for higher levels. Interferences must be assessed and corrected or the data needs to be flagged to indicate problems with the results. Interference correction must include compensation for background ions and oxides created by the plasma gas, reagents, and sample matrix.

3.0 DEFINITIONS

Definitions in text as needed or refer to the QA/QC manual or EPA method 200.8.

4.0 SAFETY

4.1 Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon tanks and fittings.

4.2 The AGILENT 7500 is fully interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled.

4.3 Always add the concentrated acid slowly to the water (not water to acid) to avoid a reaction.

4.4 *All samples handled should be considered hazardous and the appropriate PPE should be worn when running this test (safety glasses, gloves, and a lab coat).*

4.5 *Each chemical compound used in this method should be treated as a potential health hazard. MSDS for chemicals are kept at the front desk of 8100 N Austin building. Be aware of the hazardous effects of Hg and the reagents when inhaled or contacted with skin.*

4.6 *The use of laboratory equipment and chemicals exposes the analyst to several potential hazards and good laboratory technique and safety practices should be practiced at all times including the use of safety glasses, laboratory coats and acid resistant gloves when handling samples or reagents, or when in the vicinity of others handling these items.*

4.7 *Spilled samples and reagents should be cleaned up from laboratory surfaces immediately. Acidic and Alkaline spills should use spill kit "pigs" for caustic spills.*

4.8 *All additional company safety practices and procedures should be followed at all times.*

4.9 *Also refer to Chemical Hygiene plan in 8100 Conference room.*

NOTE: Please refer to the latest version of EMT' s Chemical Hygiene Plan (CHP) for more comprehensive and authoritative safety information. The information provided in this section is to be used as guidance. The information given in the CHP supercedes the information provided here.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Samples are to be collected in metal free containers such as glass quart jars or plastic pint jars with plastic or Teflon lids (jars are run periodically as samples to ensure each lot of jars is clean).

5.2 To delay the chemical and biological changes that inevitably continue after the sample is removed from the parent source, a preservation technique is required. Metal ions may precipitate as hydroxides or form complexes with other constituents. Cations or anions may change valence states under certain reducing or oxidizing conditions while other constituents may dissolve or volatilize with the passage of time. Metal cations may also absorb onto surfaces of the container (glass, plastic, etc.), such as iron and lead. Preserve the liquid samples for metals analysis to a pH < 2 with HNO_3. HNO_3 keeps the metal ions in solution and delays the ionic exchange between the metallic ions and the surface of the container.

5.3 Preserve drinking water sample with HNO_3 to pH < 2 for at least 16 hours before the analysis. All samples should be preserved as soon as possible after collection and kept on ice if not preserved. After receipt, samples are to be logged in and stored in the login cooler at 4 °C.

5.4 Even though the holding time for metals is 6 months, it is best to analyze the samples as soon as possible after collection, especially for Hg.

5.5 For dissolved samples, samples should be filtered through a 0.45-micron filter and preserved after filtration as soon as possible.

5.6 As required by the data user, field blanks should be prepared and analyzed using the same containers and acid as the samples.

5.7 All glassware used in the lab for metals should be cleaned with NOCHROMIX and/or soaked in acid and rinsed thoroughly with DI before use.

6.0 INTERFERENCE

6.1 Isobaric Elemental Interferences are caused by isotopes of different elements that form singly or doubly charged ions of the same nominal mass to charge ratio and cannot be resolved by the mass spectrometer in use. To correct this problem the data system must analyze another isotope of the interfering element and subtract the appropriate signal. This interference is uncommon and has been noted in Mo 98 (Ruthenium) and Se 82 (Krypton). Improving resolution, matrix separation, use of another isotope, or another method can be a way around the problem. Any applied corrections need to be recorded and reported with the data.

6.2 Isobaric polyatomic ion interferences (or Isobaric molecular and doubly-charged ion interferences are caused by ions having more than one atom that have the same mass to charge ratio as the isotope of interest that cannot be resolved by the spectrometer. These interferences commonly originate in the plasma or interface system from support gases or sample components (i.e. ArOH and Ar_2 with mass of 57 and 80 affect Se). Most common interferences have been identified and can be corrected for by using alternative isotopes for the analyte of interest when they occur. Equations can also be used to correct the data that must be carefully researched and reported with the data run. For instance, to correct Cd mass 114 for $Zr0^+$ contribution this correction may be derived: (m/z 114 signal)-(0.027)(m/z 118 signal)-(1.63)(m/z 108 signal) where the last two terms take into account any contribution for Sn or MoO^+ at mass 114. There may also be other factors involved while reading Cd isotopes at 108 such as contribution by $ZrOH^+$ and ZrO^+ that would need to be considered.

6.3 Physical Interferences are generally associated with sample nebulization and transport of the sample in the tubing, through the plasma, and through the plasma-mass spectrometer interface. Samples with high dissolved solids, high surface tension, and high viscosities may all be biased low as they will be "slow" to get through the system. Keeping the system clean of build-up on the torch and interface cone orifices should reduce many associated problems. To track any potentially biased low samples an internal standard is run with the samples with similar analytical behavior to the analytes of interest. This can correct for any bias.

6.4 Abundance Sensitivity is when the peaks from one mass overspread adjacent masses and contribute to that analytes signal (peak from mass next to analyte has "wings" which cross into analytes mass zone to create a biased high result). This sensitivity is affected by ion energy and the quadrupole operating pressure. Generally this problem can be corrected by adjusting the spectrometer resolution or by dilution. This is the same effect as peak broadening by ICP-AES.

6.5 Memory Interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition in the uptake system and from the buildup of sample material in the plasma torch or interface cone. Flushing the system with a rinse blank between samples can minimize memory effects. A rinse period between samples and standards long enough to flush out analytes is required. Continued memory problems warrant a cleaning of the sample uptake system, possibly including the torch and interface cones if a build-up of material is observed. If a sample following another with high results appears to have a carry-over or memory effect (first replicate high, second replicate lower, third replicate lowest) the sample should be re-run.

7.0 EQUIPMENT AND SUPPLIES

7.1 Analytical balance, capable of weighing to +/- 0.1mg, Sartorious 210S or equivalent

7.2 Argon gas supply: Industrial argon, grade 5, 99.998% purity.

7.3 Auto pipettes: In ranges from 0.1 to 10mL, to be calibrated monthly to a tolerance of 0.6% at fixed settings on the 5 and 10mL pipettes.

7.4 Hewlett-Packard AGILENT 7500 ICP-MS system (Includes AGILENT 7500 ICP-MS Instrument, ChemStation, HP LaserJet printer and Cetac ASX-510 Auto sampler)

7.5 HDPE 1-liter, 500mL, 250mL storage bottles

7.6 Printer.

7.7 Refer to individual Prep SOP's regarding digestion and preparation materials needed.

7.8 Temperature adjustable hot plate or hot block capable of maintaining 95 °C.

7.9 Volumetric flasks and pipettes. Class A.

8.0 REAGENTS AND STANDARDS

8.1 *Reagents and Standards Labeling Requirements.*

8.1.1 *Container of commercially bought standard and reagent must have label with Standard/Reagent Name, Initial Concentration, Manufacturer, Lot Number, Expiration Date, Date Received, and Date Opened.*

8.1.2 *Container of prepared solution must have label with Name of Solution, Concentration, Expiration Date and Traceability to the preparation.*

8.1.3 *Reagents and Standards Log Book must have the following information: Standard/Reagent Name, Manufacturer, Lot Number, Date Opened, Expiration Date, Initial Concentration, Initial Volume, Final Volume; Final Concentration, Initials of Person received and prepared the solution.*

NOTE: *Follow the expiration date listed on the stock reagent or listed below by prepared reagent. Re-order reagent and stock solutions as needed.*

8.2 Certified individual and custom blended standard stock solutions traceable to NIST for all analytes of interest.

8.3 DI water (ASTM type II)

8.4 Four types of blanks are required for the analysis.

8.4.1 The calibration blank is used in establishing the analytical curve. For instrument calibration and check blanks see table in Sec.17.2.

8.4.2 The method (MBLK) blank is used to assess for possible contamination resulting from the sample processing.

8.4.3 Two rinse blanks are used to flush the instrument uptake system between all samples and standards to reduce potential memory interferences.

8.4.3.1 Instrument rinse blank is 2% HNO_3 and 1% HCl to rinse out Au as needed, also helps with elevated elements. Add 20mL concentrated HNO_3 and 10mL concentrated HCl to about 500mL DI water in a 1-L volumetric flask and dilute to mark.

8.4.3.2 Auto sampler rinse station blank is *8% HNO_3 and 4% HCl* to rinse out Au as needed, also helps with elevated elements. Add 40mL concentrated HNO_3 and 20mL concentrated HCl to about 500mL DI water in a 1-L volumetric flask and dilute to mark.

8.5 Hydrochloric acid (concentrated), HCl

8.6 Nitric acid (concentrated), HNO_3

8.7 Refer to ANNEX 2 at the end of SOP for the Standards Preparation Guide.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Bi will need a second internal standard solution without Bi, using Tb instead.

9.2 Due to precipitation and instability of solutions, Au, Bi and Ta, will be prepared and kept separately for calibration and calibration check solutions.

9.3 Calibrate instrument daily starting with calibration blank (Standard# 1), proceeding through 4 multi-element standards (refer to Table 2 for the regular metals list in Sec.17.3).

9.4 The correlation coefficient for the calibration needs to be > 0.995 for all target elements. *If the coefficient is lower for any of elements of interest, the second or fourth calibration point may be excluded from the calibration curve.*
Calibration must include a calibration blank and at least 1 additional calibration point for each element, which brackets the expected sample analyte concentration range.
If the coefficient is still lower for any of elements of interest, the instrument needs to be re-calibrated for all elements included in the run before any analysis can begin. If calibration fails again, problem needs to be investigated and corrected prior to analysis. There could be contamination in the blank, one of the standards may be diluted wrong, or the instrument uptake system may not be functioning properly (the last two should effect all elements, a contamination or memory effect may only effect one element).

9.5 Calibration curve calculation, see Sec.11.1.

9.6 The Initial calibration verification (ICB and ICV) is done immediately following the initial calibration. *ICV, midpoint standard (100 ppb trace, 50 ppb Be, and 500 ppb minerals) is prepared from the standard unassociated with the source of calibration standards.* To be acceptable an ICB should be < 3 times the IDL. The recovery of the ICV must be within 90 -110%.

9.7 The initial calibration blank, ICB, 2% HNO3, should be analyzed immediately following calibration.

9.8 The continuous calibration verification, CCV, should be analyzed at the start of the run, as it is the mid-point calibration check and after every tenth injection/sample The CCV should also be run at the end of the sample run.

9.9 Continuing Calibration Blank (CCB): must to be run every 10 injections during run and at the end of the run.

10.0 PROCEDURE

NOTE: Also see ANNEX 1 at end for run order.

10.1 Instrument Set-Up Prior to Run

10.1.1 Turn on hoods and water re-circulator systems, verify Argon pressure, bring up the computer software, verify rinse solution and internal standards full, check that drain bottles not over-flow, and start the instrument (see below).

10.1.2 Load instrument software from "ICP-MS Top" icon on main screen, select "Methods" from toolbar, go down to "Load" and select method (COMBONEW.M or EPRODUCT.M if Bi, Au and Ta are elements of interest), and select linked calibration internal standard table (COMBONEW.C or EPRODUCT.C).

10.1.3 To move auto-sampler to 2% rinse, go from "Instrument Panel" select "ALS" and click position of solution (generally position 1). Ensure internal standard line in 2% rinse bottle.

10.1.4 Go to "Maintenance" from "Instrument Panel" and select "Sample Introduction".

10.1.5 Go to "peri pump", and in box enter a value from 0.1 to 0.3 to turn on pump. Connect tubings and clamp onto pump and monitor uptake rate for consistent flow.

10.1.6 Close "Sample Intro" screen that will turn off pump before proceeding to ignition of the plasma.

10.1.7 To start instrument (go from "Standby" to "Analysis" that will load operating conditions based on most recent saved tune file) go to ICP-MS "Top" menu, "Instrument", then "Instrument Control", then "Plasma On" and wait for ignition and stabilization. Wait a minimum of a half an hour before analysis or tuning.

10.1.8 A clog can be checked for by monitoring the tuning solution responses, cleaning of the torch, nebulizer, or cones may be needed.

10.1.9 Check mass calibration, stability, and resolution checks for the analyte mass regions by tuning the instrument prior to the calibration with the Agilent instrument tune solution checking masses of Li, Y, Ce, Tl, and Co at 10 ug/L.

10.1.10 Move auto sampler to solution (from "ALS" procedure) and from ICP-MS "Top Screen" toolbar go to "Instrument", then "Tune" then "Tune Sensitivity", then "Start". Refer to Agilent ICP-MS Chemstation Operator's manual page 4-12 or initial set-up tunes in binder for guideline on lens and plasma settings for environmental analysis. Ce used mainly for Oxides, normally monitors the three masses for Li (7), Y (89), and Tl (205). Masses can be changed to monitor internal standards or memory from samples by going to "Acq.Params" from the Tune screen and changing the masses to monitor. Must always stop the scrolling reading before any changes or exiting is allowed. Can monitor rinse-outs of high elements by monitoring their associated masses (after high Na samples monitor the mass 23 for Na rinse out).

NOTE: Do not use regular Agilent tuning solution for "P/A" (pulse to analog) tune. Must use a solution containing all masses at approximately 200 ug/L or higher if sensitivity listed as too low for that element in the tune report.

10.1.11 Generate the tune report for the regular daily tuning by clicking the "Tune Report" icon at the top of the screen, allowing the instrument to run all of the check parameters, and allowing for the report to print. This is not the method tune-that will be carried out by the instrument in the sequence control prior to calibration. Can increase pump from tune screen to get solutions into instrument faster.

10.1.12 Set internal standard tubing into internal standard solution and begin flow into the system. Monitor the internal standard flow from the "Tune" screen by selecting for the masses in the tuning solution (6 for Li, 45 for Sc, 72 for Ge, 89 for Y, 159 for Tb [e-products], and 209 for Bi). Can only monitor 3 masses on the tune screen at one time. Once internals into system

and look stable, one can proceed. Make sure you use the correct solutions for the methods, as the e-product method cannot contain the Bi in the internal standard.

10.1.13 Set the batch run defaults from the last sequence run for "TUNE" block (use Tune1 for type). Set calibration in "CALIB" block and for CCV and CCB checks every 10 samples in "CCV" block. For night runs, select "TERM" block for shutdown at end of sequence blocks instead of "CCV" making sure that "Keyword" is the sample type and "Standby" is selected for the method (do not need other columns).

10.1.14 To set up run (for samples), edit the "SAMPLE" block section for the sequence if staying with the same method from last run. Need to re-edit all and change the standards and method codes for above blocks if switching or running multiple methods.

NOTE: Must always select, modify, and re-name the last sequence run to set up the new sequence or the software will not recognize.

10.1.15 From main screen go to "Edit Sample Log Table" icon, select most recent run sequence and edit "SAMPLE BLOCK" (can also go from ICP-MS "Top" screen toolbar to "Sequence" and "Load" if instrument in "Analysis" mode (plasma on).

10.1.16 Type in sample type: AllRef for un-spiked and undiluted sample, MBLK for prep blank, LCS for Lab control spike, Sample for regular non-QC samples, *MS* for spike, MSD for spike duplicate, *Dil for further 1:25 dilution test*, and Dup1 for duplicate check. (Done by 1 click in column and selecting from list).

10.1.17 Type in vial numbers (start at 2101 for first row, highlight column down, and use "Fill Down" option from right clicking mouse to auto increment all samples.)

10.1.18 Double click cell in Method column and select "COMBONEW.M" or "EPRODUCT.M" from the list and use "Fill Down" for all samples.

10.1.19 Type in the sample information or comments into the Sample column: work order numbers, sequence from prep, etc.

10.1.20 Type in Dil/ Lvl dilution factors in this column (*1 for all samples, blanks, LCS and 5 for DIL1).*

NOTE: Must fill in these five minimal columns for sequence to run properly and make sure to SAVE changes by renaming file with the day's run date.

10.1.21 Select "Whole Block" to view when completed to check for typos (which can stop the run) and print out as the run log.

10.2 Instrument Run

10.2.1 From ICP-MS "Top" screen go to "Sequence", then "Load and Run sequence", then select sequence needed (created above) from list, then "OK", then click "Run Sequence" to start the analysis.

NOTE: Run can be stopped at any point by clicking "abort button" or "stop run" on display during run. Can re-start by loading the sequence table under "Position sequence and Run" and highlighting the sample to start the run on and hit "OK". Must re-start sequence with Calibration blank to re-set the Internal Standard true values. It is better to add to Sample block during run than to stop the sequence).

10.2.2 Load the method TN2008.m. for tuning and generate the tune report (should be run by sequence prior to calibration if set correctly). Mass calibration and resolution parameters must be met prior to analyzing samples by running the 200.8 Tune check solution (refer to QC for tuning). Failures must be addressed by checking manufacturer tuning solution and adjusting torch or lens parameters.

10.2.3 Calibrate the instrument starting with the blank and proceeding with the standards. Uptake time should be set for at least 30 seconds and rinse out for 1 minute. Must use three integrations for analytes.

10.2.4 To print the calibration summary report go to: "Offline Data Analysis" (from main screen), "File", "Load" (current run, should be selected), then to "Calibrate" and "Print Summary Report". Will have all masses intercepts and correlation coefficients. (Offline data Analysis can be used to reprint and monitor the current run or check other past runs for reference).

10.2.5 Proceed with ICV, and ICB at the beginning of the run. Insert CCV and CCB at the rates of one set per 10 injections.

10.2.6 All masses that could affect data quality need to be monitored to determine potential effects from matrix components on the analyte peaks. Interfering masses should be monitored in the same scan as used for the collection of the data. See also ICS solution in QC portion of SOP. All interference corrections needed should be loaded and set to "on" prior to the start of the sequence run start.

10.3 Wastewater Total Metals

10.3.1 Unlike the drink water and dissolved samples (see below), the digested wastewater samples can be put directly onto the instrument after a 1:5 dilution and run after sample prep is complete. Refer to digestion methods and SOP's regarding the prep of wastewaters for digestion (method 3015, SOP 101).

10.4 Drinking Water

10.4.1 To determine whether digestion of the sample is required, the turbidity of the acidified sample must be measured using an approved method and only after preservation is complete (SOP 064). Preservation is complete after the acidified sample has been held for 16 hours. Before sample processing is started, sample pH must be verified to be less than 2. If pH greater than 2, acidify sample to < 2 and wait 16 hours to do turbidity screen. Document the pH of the sample in the turbidity book (refer to SOP 064 and turbidity methods).

10.4.2 For the "direct analysis" of total recoverable analytes in drinking water samples containing turbidity <1 NTU, add an appropriate volume of (1+1) HNO_3 to an unfiltered acid preserved sample aliquot to adjust the acid concentration of the aliquot to approximate a 2% (v/v) HNO_3 solution (add 2mL concentrated HNO_3 to 99mL of sample). Allowance for sample dilution should be made in the calculations.

10.4.3 For the determination of total recoverable analytes in drinking water samples with >1 NTU turbidity:

10.4.3.1 Transfer 100mL aliquot from a well mixed, acid preserved sample to a 250mL Griffin beaker (smaller sample aliquot volumes may be used when necessary. Set up additional beakers for blank, fortified blank, sample spike and spike duplicate.

10.4.3.2 Add 2mL (1+1) HNO_3 and 1.0mL of (1+1) HCl to the beaker.

10.4.3.3 Place the beaker on the hot plate for solution evaporation.

10.4.3.4 Reduce the volume of the sample aliquot to about 20mL by gentle heating at 85 °C uncovered and 95 °C covered with a watch glass.

10.4.3.5 Reflux the sample for additional 30 minutes on the hot plate and allow the beaker to cool.

10.4.3.6 Quantitatively transfer the sample solution to a graduate cylinder and bring the volume to 50mL with DI water (bring to 25mL of the volume if low level needs to be achieved).

10.4.3.7 Filter the sample if suspended solid is present.

NOTE: All drinking waters samples (run with digestion or without digestion) are to be run with dilution 1:1

10.5 Dissolved Metals

10.5.1 For the determination of dissolved analytes in ground and surface waters, unpreserved sample should be filtered through 0.45um membrane filter. Preserve the samples with nitric acid to match the matrix of the calibration standard only after the filtration. Add 5mL of concentrated HNO_3 to 45mL of sample to matrix match and analyze. Allowance for sample dilution should be made in the calculation.

10.5.2 To reduce potential interferences, dissolved solids should be <0.2% (w/v).

10.5.3 If physical interferences are present they must be reduced by diluting the sample

10.5.4 Salt buildup at the tip of the nebulizer due to high dissolved solids affects aerosol flow rate and causes instrumental drift. This could also be controlled by diluting the samples high in salts and dissolved solids.

10.5.5 If a precipitate is formed during acidification, transport, or storage, the sample aliquot must be digested according to Drinking Water digestion method.

10.5.6 Use Drinking Water digestion method if low level needs to be achieved.

NOTE: All samples digested need 1:5 dilution to matrix match acid to 2% concentration and reduce interferences. Dissolved and Drinking water samples need to be matrix matched to 2% HNO_3 prior to analysis.

10.5.7 Calculate and evaluate QC data using "ChemStation software to make sure all data within specified acceptance ranges. Report data with any flags as needed into computer. All calculations will be performed and reported out with flags by the "generate custom reports" in Chemstation.

10.5.8 Ensure the CCV and CCB are run and within acceptance limits.

10.5.9 Monitor internal standard during run to ensure values do not deviate from the acceptable ranges. Re-tune and start run over if needed. Software will monitor and post all internal standard results at bottom of page for all samples.

10.5.10 If the sequence for method is set properly all of the above steps from 10.2.5 to 10.2.11 including calculations will be performed, reported out and data will be stored by the Chemstation software).

10.6 Shut-Down

10.6.1 After sample run is complete and final QC checks are verified to be acceptable, allow instrument to flush out any sample residue for about 15 minutes minimal. By alternating between rinses such as 2% HNO_3 with 1% HCl and DI water with auto sampler most residues should be removed. Allow longer time if samples with high levels of analytes were run. Remove tubing from internal standard reservoir and place in 2% HNO_3 during this process to rinse lines (and help clear out Li which is "sticky" on the cones and lenses). Always leave

liquid in internal standard line or it will need to be re-primed. Monitor masses that were high in the tuning screen to make sure properly rinsed out of the system (Mineral masses or Li).

10.6.2 Shutdown and turn off instrument by going to instrument control screen and clicking plasma off (usually left in "stand- by" mode). When plasma is off and the instrument switches from "Analysis" to "Standby" mode, move auto sampler probe to "Home" position to turn off reservoir rinse pump. Disconnect pump tubings to avoid crimping and extend lifetime of tubing.

10.6.3 Turn off water re-circulator and hood after instrument heat dissipated.

11.0 CALCULATIONS

11.1 Calibration Curve calculation:

$$Y = aX + [b]$$

11.2 Sample final results calculations from the ICP-MS may be calculated as follows and reported in the appropriate units and if required for solids, dry weight correction will be done by Omega (most all calculations on run will be done by the Chemstation software):

Final concentration of analyte (ppm): **= (A * B * C) / (mL of sample x 1000)**

Where:
A = Instrument reading off calibration curve (in ug/L)
B= Final volume of digested sample (if digested) in mL.
C= dilution factor after digestion

11.3 Spike calculation (% recovery):

$$\% \text{ Recovery} = \frac{\text{Spiked sample - unspiked sample}}{\text{True value of Spike}} * 100$$

NOTE: On spiked sample if < MDL, do not need to subtract off value from spiked sample.

11.4 Relative Percent Difference (RPD) calculations:

$$\% \text{ RPD} = \frac{\text{Abs (MS – MSD)}}{\text{Ave (MS+MSD)}} * 100$$

11.5 The following interference equations are used to correct for isobaric elemental and polyatomic interferences. All equations must be specified in the ChemStation method before any other data acquisition or data analysis parameters are set.

11.6 The correction equation for ^{44}Ca must be determined empirically whenever instrument tune conditions or matrix composition change significantly. A Ca and Sr free blank and a Ca free Sr standard (ca. 100 ug/L) are required for this determination. First, measure and record the counts for the blank at m/z 44 (background CO_2). Second, measure the 100 ug/L Sr standard at m/z 88 and 44. The correction factor for ^{44}Ca is calculated using the following equation.

$$\text{Correction factor} = \frac{Sr^{++}\text{(m/z 44) counts – Blank (m/z 44) counts}}{^{88}Sr \text{ counts}}$$

11.7 Calculate analytical results:

$$C_x = \frac{A_b V_s C_s}{(A_a - A_b) V_x}$$

Where:
C_x, =Concentration of the sample.
A_a,= Analytical reading of sample with spike.
A_b, =Analytical reading of sample without spike.
V_s, =Volume of the spike added.
V_x, =Volume of the sample aliquot.

C_s, =Concentration of the spike.

11.8 *Calculation of the common quality control parameters (e.g., percent recovery, percent difference, relative percent difference (RPD), and relative standard deviation (RSD)), are given in the EMT Laboratory Quality Assurance Manual.*

12.0 QUALITY CONTROL

12.1 Batch QC

12.1.1 **Control limits:** Generate and systematically update control limits for LCS % recovery, MS/MSD % recovery and RPD %. Use in house generated control limits for data evaluation if the limits are more stringent than the pre-set. After collection of *15-20* points Inspect control charts visually for indication of systematic trends.

12.1.2 **Laboratory Control Sample (LCS):** At least one LCS should be analyzed with each batch of 20 samples or fewer. The recovery of any analyte of interest must be within 85-115% or within established control limits (whichever is more stringent). Batches with a LCS outside limits will need to be re-digested and re-analyzed, *unless LCS fails high and sample is non-detected for the analytes of interest.* A failed LCS could be a prep problem or indicative of instrument drift. The problem needs to be investigated and documented.

12.1.3 **Low Level Check standard** (Performance Evaluation Mix (PEM) sample type): is analyzed to verify the instrument performance near or at the reporting limit. Prepare the standard by diluting the CCV 100 times. A low level check should be run with each analytical batch with an acceptance of +/- 30%. May re-run with fresh solution if contaminant or potential memory effects occur.

12.1.4 **Matrix Spike (MS), Matrix Spike Duplicate (MSD):** Analyze MS/MSD only for samples that contain < 0.1% of target analysis. Spiked samples shall be rotated among clients' samples. Acceptance range for spikes recovery is 70-130%, or within laboratory established control limits (whichever is more stringent). If MS/MSD is out of range, then the samples need to be checked for memory, or background interferences through the use of dilution test, post-digestion spike (PDS) or method of standard addition (MSA).

12.1.4.1 **Dilution test**: Dilute sample and MS/MSD 1:5 and check for recovery between 75 and 125% on diluted MS/MSD. The concentration of the original and diluted sample should agree within 10% of the original value. If the difference is greater than 10%, matrix interferences are suspect and should also be shown and confirmed by the internal standards in the affected analytes mass region.

12.1.4.2 **MSA: Method of Standard Additions** - The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, the aliquot should be prepared so that the resulting concentrations are approximately 50, 100, 150% of the expected concentration of the analyte. The concentration of each solution is determined and then plotted on the vertical axis of a graph, with the concentration of the known standards plotted on the horizontal axis. When the resulting line is extrapolated, the point of interception of the abscissa is the concentration of the analyte in the sample. The curve should be linear and have at least 0.995 coefficient correlation. The slope of the MSA plot should be within 20% of the standard curve to be accepted. (An example plot is shown in fig. 1.The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, a known concentration of an analyte is added to one and

a diluent to another. The analytical results are determined and calculated as seen in Sec.11.7.

12.1.4.3 **Post-digestion spike:** Add analyte spike to a portion of digested sample or its dilution. The concentration of added analyte should be 0.5 to 2 times of sample background. If PDS is not recovered within 75-125% of the true value a matrix effect is possibly occurring and further dilution may be needed to compensate for matrix effect.

12.1.5 **Method Blank (MBLK):** At least one MBLK should be analyzed per batch of 20 samples or fewer. When MBLK values exceed 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL (whichever is greater), the samples needing the failed analyte should be re-digested and reanalyzed unless the samples needed are non-detect for that analyte. A failed blank may indicate a baseline drift or contamination. Continuation of a problem will need to be investigated and documented. The method blank must contain all the reagents and in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis (refer to the metals digestion methods).

12.1.6 **MS/MSD**: A control limit for RPD is 20% or within established control limits (whichever is more stringent). Samples outside of range may be re-checked with a dilution test in case of suppression or background. If RPD is still outside of range, the problem needs to be investigated further. The problem might relate to the sample uptake system or it might be a digestion error. If the problem relates to the digestion batch, the whole digestion batch might need to be re-digested and re-run. Otherwise re-digest the sample with the failed RPD as the problem is most likely matrix related to the specific sample. If second digestion fails, data may be reported with a flag with a description of the problem.

12.1.7 **Routine Dilution test** should be run with each batch or 20 samples. The test is performed on samples with concentration of element(s) at least 50 times higher then MDL. Dilute sample fivefold (1+4). The original results and the results on diluted sample should agree to within 10 % of the original determination. If not, matrix interference is suspected. The sample should be re-checked or flagged for affected analytes.

12.2 **Linear dynamic (or calibration) range (LDR):** Linear ranges are detector limited and the upper limit should be established for each analyte by determining the signal responses from a minimum of three different concentration standards with one being near the upper limit. The upper LDR limit should be an observed signal no more than 10% below the level extrapolated from the lower standards. Care MUST be used when determining upper limits as damage can occur by overwhelming a detector with high levels of analytes that could potentially destroy or cause irreparable damage to the detector. Samples over 90% of established upper limit must be diluted and re-analyzed (Chem. station will monitor and flag for over-range elements). LDR should be performed whenever significant changes in the instrumentation are introduced. A low level standard near LOQ (and reporting limit) needs to be included with the LDR study to prove linearity at the low end of the calibration.

12.3 **Instrument detection Limit (IDL):** Determine IDL by running a blank solution 7 times on three non-consecutive days and calculating the standard deviation (SD) for each run. The average of SD from the 3 different days results is the IDL. The IDL needs to be run before anything else on a new instrument and should be updated every three months as assurance that the sensitivity of the instrument is not changing over time.

12.4 **Instrument Performance Check**

12.4.1 **An Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)**: The results should be < 3 times the IDL. The ICB needs to be run at the start of run following the ICV. A CCB needs to be run every 10 injections during run and at the end of the run. If ICB or CCB is greater than +/- 3 x IDL in the first attempt, re-analyze with fresh aliquot or after a rinse if

carry-over is suspect. If it is still out, check the tuning, check for clogs, and check the internal standard results. Correct the problem, document, re-tune or calibrate as needed. If CCB failed during the run, prior sample section needs to be re-analyzed for affected elements unless the samples are non-detect.

12.4.2 Initial Calibration: Refer to 9.3, 9.4, and 9.5.

12.4.3 **The Instrument Calibration Check Solution (ICV and CCV): ICV** should be done immediately following calibration with an acceptance of +/- 10% of the true value (TV). *ICV must be from a different source than the calibration solutions and near the midpoint of the calibration curve (100 ug/L trace, 50 ug/L Be, and 500 ug/L minerals).* If ICV fails in first attempt, an immediate second analysis of the ICV needs to be done to confirm unacceptable performance. If ICV fails the second time check tuning, then check for clogs, check internal standard results, correct the problem, document, re-tune or calibrate as needed and re-start the run. Elements with failed ICV can't be analyzed. *CCV must be from the same source as calibration solutions and near the midpoint of the calibration curve (100 ug/L trace, 50 ug/L Be, and 500 ug/L minerals).* The CCV check solution should be run after every 10 injections and at the end of the run with an acceptance of +/- 15% of the true value (TV). If CCV fails in the first attempt, then check the tuning, check for clogs and check the internal standard results. If a known reason is found and can be documented (probe contamination, carry-over, etc.), then document the run and re-run CCV two more times. If the re-runs of CCV fails low, correct the problem, document it, re-tune or calibrate, as needed and prior sample section needs to be re-run for failed analytes. If initial check or re-runs of CCV fails high, may report elements in the section as long as they are non-detected or less than reporting limit. Otherwise samples need to be re-analyzed after problem corrected. A CCV following samples with high viscosities may be suppressed. Check for low internal standard results, and re-checked once after allowing for a rinse-out. Document all problems on the run. If passing on the re-check, prior samples should be re-run for failed analytes with dilution or checked with a dilution test or bench spike to check for matrix effects.

12.5 Internal Standards (IS or ISTD as sample type):

12.5.1 A known value is to be pumped into instrument at same time and rate of samples and combined at the pump to ensure an even and mixed rate. The mixed solution should be approximately 50 ppb of: Li 6 (low mass monitoring), Sc 45 (Be to Fe mass corrections), Ge 72 (Co to Se mass corrections), Y 89 (Mo to Ag mass corrections), Bi 209 (Hg to Pb mass corrections), Tb 159 (Ba & Sb mass corrections if Y is present in sample)

12.5.2 Internal standard responses need to be continually monitored and within 60 to 125% of the absolute response given by the instrument blank. If IS falls outside the 60 to 125% range for a sample, recheck the blank after rinsing the system. If the response is acceptable on the blank recheck, re-run the sample with a dilution as matrix problems probably are responsible. If the response is still out, check the tuning of the instrument and retune and start run over as needed. If it is outside of the range on a sample, assume matrix interference and dilute sample 1:5 and re-analyze.

12.5.3 If IS is out on a QC sample or standard, check tubing line for bubble or problem and check blank solution response. One may need to re-calibrate and re-analyze affected section of run.

12.5.4 Very high IS recoveries may mean that no sample went through (air) or the probe may have missed the sample.

12.5.5 If a sample contains one of the internal standard elements one may re-select the IS for the affected elements being monitored by that element to another IS element within 50 mass units in the Calibration Table "IS Select" column from "Offline Data Analysis". The calibration, QC samples, and affected samples need to be re-processed and re-calculated in the "Do List".

12.6 **Tuning Check Solution (for 200.8):** This solution is used to verify instrument tune and mass calibration prior to analysis. The solution contains Be, Mg, Co, In, and Pb in 2% HNO_3 at a concentration of 10 ppb for each element. Low mass can be determined by using Magnesium isotopes 24, 25, and 26. High mass can be determined by using Pb isotopes 206, 207, and 208. Resolution should be < 0.75 amu at 5% peak height. Check instrument stability by running tuning solution 5 times and verifying standard deviation (SD) < 5% for all analytes absolute signals. Elements should be < 0.1 amu from the true values.

13.0 REPORTING

For reporting refer to the most current revision of SOP # 209.

14.0 DEVIATIONS FROM THE REFERENCE METHOD

14.1 Using a spiked duplicate sample over a sample duplicate.

14.2 Samples with non-detect analytes and associated with method blank > 2.2 times MDL can be reported.

14.3 Analyzing a larger list of metals than originally listed in the method.

14.4 MDL may be based on IDL of 200.8, 6020A, or MDL. Whichever is highest is used for MDL and PQL determination to ensure value is not unreasonably low.

14.5 Rinse blank is 1% HCl and 2% HNO_3 to ensure against memory effects and to rinse out Au, Ag and other "sticky" elements more effectively.

14.6 Using post digestion spikes for dissolved and Drinking water samples without digestion as well as matrix interference checks for problem samples.

14.7 Se is working properly at same level as trace metals (not set to 5 times higher and have not experienced any problems).

15.0 METHOD PERFORMANCE

15.1 All files for the IDMP's, MDL's, LDR's, and IDL's are tabulated and stored on the network drive directory of "L" in the file "&Exceldoc" under each of their representative instrument and file type headings.

15.2 The control charts are put into a tabulated spreadsheet after creation from LIMS system and stored also on the "L" drive in the "&Exceldoc" folder in the subfolder "control limits" for easy reproduction and analyst review.

15.3 Initial Demonstration of Capacity (IDC): The initial demonstration of performance is used to characterize instrument performance and laboratory performance prior to analyses conducted by this method. IDC should be performed whenever a new analyst is trained or significant changes in the instrumentation are introduced. The analyst must run four digested standards for all elements to be analyzed on the instrument at a concentration of approximately 10-50 times MDL or IDL (instrument detection limit). The % RSD needs to be less than 15% and each replicate recovery should be within 80-120%.

15.4 Method detection limit (MDL): MDL studies should be performed annually and the reporting limit should be determined accordingly. Reporting limit is generally 3 times the MDL (see reporting section). Determine MDL by running a set of 7 digested standard solutions for all elements being

analyzed at a concentration of about 1-5 times prior MDL or IDL and the resulting standard deviation (SD) is multiplied by 3,14. If calculated MDL is lower than the IDL, the IDL value is considered the MDL. If the MDL result comes out lower than 10% of the standard run, use the standard value as the minimum detection level as long as limit meets all needs of the clients. All digestates will be run at 1:5 dilution for MDL calculation. Acceptance criteria for MDL study can be found in SOP# 218

15.5 *Method performance is detailed in the EMT Quality Assurance Manual, which includes sections on Method Startup, Reporting Limits, Method Detection Limits (MDLs), Method Control, and Initial Demonstrations of Capability (IDC).*

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 Dispose of all acid wastes (samples, expired standards, rinse solutions, etc.) in the HNO_3 waste acid container located on the counter at the rear entrance of 8100 N. Austin. A specialized waste hauler then picks up the waste.

16.2 Clean up all spills of acid properly by neutralizing the spill and disposing of it.

16.3 Old stock standards and solution containers to be disposed of should be put into the acid waste container and rinsed generously 3 times with water before being thrown away. Many of the polypropylene containers can be recycled after rinsing.

16.4 *Refer to EMT "Quality Manual" for samples disposal used in this SOP.*

17.0 TABLES, DIAGRAMS, FLOW CHARTS, INSTRUMENT MAINTENANCE

17.1 Elements

Element		Chemical Abstract Services Registry Numbers (CASRN)
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-39-3
Bismuth	(Bi)	7440-69-9
Boron	(B)	7440-42-8
Calcium	(Ca)	7440-70-2
Cadmium	(Cd)	7440-43-9
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Gold	(Au)	
Lead	(Pb)	7439-92-1
Manganese	(Mn)	7439-96-5
Mercury	(Hg)	7439-97-6
Magnesium	(Mg)	7439-95-4
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Palladium	(Pd)	
Platinum	(Pt)	
Phosphorous	(P)	7723-14-0

Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Strontium	(Sr)	7440-24-6
Tantalum	(Ta)	
Tellurium	(Te)	13494-80-9
Thallium	(Tl)	7440-28-0
Tin	(Sn)	7440-31-5
Titanium	(Ti)	7440-32-6
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

17.2 Instrument Calibration, and Check Blanks (ICB, CCB)

Compound/Standard:	Initial conc.:	mL used:	Total volume (mL):	Final Conc.:	Holding Time
HCl	Conc.	10		1%	One Month
HNO_3	Conc.	20	1000	2%	One Month

17.3 Table 2 Calibration Standard Summary (COMBONEW Method)

Elements:	*Blank*	*Standard 1*	*Standard 2*	*Standard 3*
Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Sn, Ag, Tl, V, Zn	*0*	*22 ug/L*	*110 ug/L*	*220 ug/L*
Be	*0*	*11 ug/L*	*55 ug/L*	*110 ug/L*
Ca, Fe, Mg, P, K, & Na	*0*	*55 ug/L*	*275 ug/L*	*550ug/L*

17.4 Elements

Element	**Interference Correction Equation**
^{44}Ca	(1.000)(44C)-(0.0271)(88C)
^{51}V	(1.000)(51C)-(3.127)(53C)+(0.353)(52C)
^{75}As	(1.000)(75C)-(3.127)(77C)+(2.736)(82C)-(2.760)(83C)
^{82}Se	(1.000)(82C)-(1.01)(83C)
^{98}Mo	(1.000)(98C)-(0.146)(99C)
^{111}Cd	(1.000)(111C)-(1.073)(108C)+(0.764)(106C)
115(In)	(1.000)(115C)-(0.016)(118C)
^{208}Pb	(1.000)(208C)+(1.000)(207C)+(1.000)(206C)
^{6}Li	(1.000)(6C0-(0.08)(7C)

17.5 Routine maintenance should be performed on each instrument according to the manufacturer's instructions.

17.6 Preventive maintenance should include: checking of pump tubings, inspection of torch and injection tube, nebulizer changing or cleaning along with spray chamber, check vacuum oil level and filter, check water chiller levels and filter, and checking of interface and skimmer cones for buildup. Document all the actions in the maintenance book. More frequent maintenance should be done if necessary. Refer to manufacturer's manual book and inline maintenance "clock" which will bring up messages regarding needed maintenance to be performed after so many hours of use.

17.7 Refer to ANNEX 1 for ICP-MS Run Order

17.8 Refer to ANNEX 2 for Working Tuning Check Solution and Internal Standard Solution- Preparation Guide

17.9 Refer to ANNEX 3 for Calibration Standards and Instruments Check Standards for the regular metals list- Preparation Guide

17.10 Refer to ANNEX 4 for Calibration Standards, Instrument Check standards and Working Internal Standard for additional metals list (Bi, Au and Ta as target elements)- Preparation Guide

17.11 Refer to ANNEX 5 for ICP-MS Quality Control Samples with Limits and Frequencies"

18.0 REFERENCES

18.1 Analysis of Drinking Water and Waste Water by ICP-MS Using EPA Method 200.8, Agilent Application Note, April 2000.

18.2 EPA 200.8 Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma-Mass Spectroscopy, Agilent SOP Draft Revision C.00.01, October 1999.

18.3 *Laboratory Quality Assurance Manual, Environmental Monitoring & Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.*

18.4 US EPA Method 200.8, Determination of trace elements in waters and wastes by ICP-Mass Spectrometry, Revision 4.4, April 1991

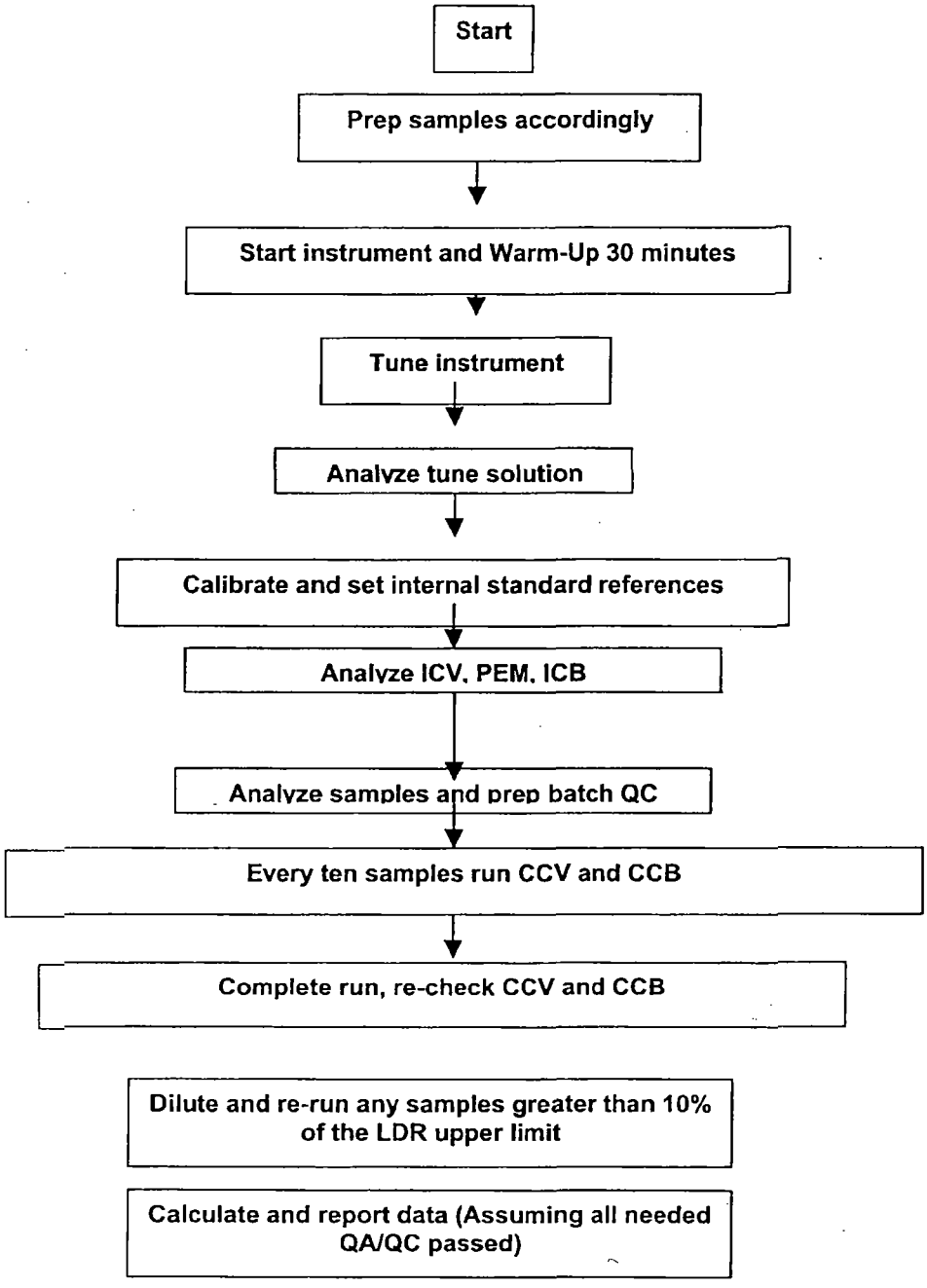
18.5 US EPA method 200.8, Determination of trace elements in waters and wastes by ICP-Mass Spectrometry, Revision 5.4, May 1994.

18.6 US EPA method 200.8, Determination of trace elements in waters and wastes by ICP-Mass Spectrometry, Revision 5.5

18.7 User Manuals for Agilent 7500 ICP-MS

ANNEX 1

ICP-MS Run Order:



ANNEX 2

Working Tuning Check Solution and Internal Standard Solution- Preparation Guide

Working Tuning Check Solution:

Compound/Standard:	Initial conc.(mg/L):	mL used:	Total volume (mL):	Final Conc. (ug/L):	Holding Time
Custom tune solution: Be, Mg, Co, In, Pb	1	2	200	10	One Month
HNO3	conc.	4		2%	

Working Internal Standard Solution:

Compound/Standard (from individual solutions):	Initial conc.(mg/L):	mL used:	Total volume (mL):	Final Conc. (ug/L):	Holding Time
Sc, Ge, Y, & Tb	100	2.5		500	
Bi, Li (6)	1000	0.25	500	500	One Month
HNO_3	Conc.	10		2%	

ANNEX 3

Calibration Standards and Instruments Check Standards for the regular metals list- Preparation Guide

Working Calibration Standards:

Intermediate Calibration Standard

Compound/Standard:	*Initial conc.(mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
In CPI Custom Blend: *Be* ***Trace metals**: Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Ti, V, Zn, Sn, Tl* ***Minerals:*** *Ca, Fe, Mg, P, K, Na*	*50* *100* *500*	*10*	*100*	*5000* *10000* *50000*	*2 weeks*
HNO_3	*Conc.*	*2*		*2%*	

Working Standard # 1 is the 2% HNO_3 instrument blank

Working Standard # 2

Compound/Standard:	*Initial conc.(ug/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
In Intermediate Calibration Standard *Be* ***Trace metals**: Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Ti, V, Zn, Sn, Tl* ***Minerals*** *Ca, Fe, Mg, P, K, Na*	*5000* *10000* *50000*	*1.1*	*500*	*11* *22* *55*	*2 weeks*
HNO_3	*Conc.*	*10*		*2%*	

Working Standard # 3

Compound/Standard:	*Initial conc.(ug/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
In Intermediate Calibration Standard *Be* ***Trace metals**: Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Ti, V, Zn, Sn, Tl* ***Minerals*** *Ca, Fe, Mg, P, K, Na*	*5000* *10000* *50000*	*5.5*	*500*	*55* *110* *275*	*2 weeks*
HNO_3	*Conc.*	*10*		*2%*	

Working Standard # 4

Compound/Standard:	*Initial conc.(ug/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
In Intermediate Calibration Standard *Be* ***Trace metals**: Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Ti, V, Zn, Sn, Tl* ***Minerals*** *Ca, Fe, Mg, P, K, Na*	*5000* *10000* *50000*	*11*	*500*	*110* *220* *550*	*2 weeks*
HNO_3	*Conc.*	*10*		*2%*	

NOTE: Solutions are to be prepared every two weeks.

Working Instrument Check Standard (ICV)
(The ICV value is near the midpoint of the calibration, but other than the calibration point. The standard is prepared from one custom blend solution, different source than calibration.)

Compound/Standard:	*Initial conc. (mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Sn, Ag, Ti, Tl, V, & Zn	*50*	*1.0*		*100*	
Ca, Fe, Mg, P, K, & Na	*250*	*1.0*	*500*	*500*	*One Month*
Be	*25*	*1.0*		*50*	
HNO_3	*Conc.*	*20*		*2%*	

Working Instrument Check Standard (CCV)
(The CCV value is near the midpoint of the calibration, but other than the calibration point. The standard is prepared from one custom blend solution, the same source as calibration.)

Compound/Standard:	*Initial conc. (mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Sn, Ag, Ti, Tl, V, & Zn	*50*	*1.0*		*100*	
Ca, Fe, Mg, P, K, & Na	*250*	*1.0*	*500*	*500*	*One Month*
Be	*25*	*1.0*		*50*	
HNO_3	*Conc.*	*20*		*2%*	

Low Level Check Standard (prepare from CCV check standard)

Compound/Standard:	*Initial conc. (ug/L):*	*mL used:*	*Total volume mL*	*Final Conc. (ug/L):*	*Holding Time*

Al, Sb, As, Ba, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Sn, Ag, Ti, Tl, V, & Zn	*100*	*0.1*	*10*	*1.0*	*One Day*
Ca, Fe, Mg, P, K, & Na	*500*			*5.0*	
Be	*50*			*0.5*	
HNO_3	*2%*	*2*		*2%*	

Bench Spike / Post Digestion Spike (PDS):
Will be using dilution of the CCV standard, generally a 1:1 dilution of the CCV to a pre-diluted aliquot of the sample.

ANNEX 4

Calibration Standards, Instrument Check standards and Working Internal Standard for additional metals list (Bi, Au and Ta as target elements)- Preparation Guide

Intermediate Calibration Solution 100 ppm for: Au, Bi, Pd, Pt, Sr, Te (Ta from single standards make separate intermediate for final solution due to higher level): To a clean vessel measure 4 mL of 2% HNO_3 and 1 mL 1000 ppm of each of the above elements. Mix well. Prepare fresh when needed. For Ta, measure 9 mL of 2% HNO_3 and 1 mL of Ta. Mix well. Prepare fresh when needed.

Working Calibration- Standard # 4:

Compound/Standard:	*Initial conc.(mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
Intermediate of: Au, Bi	*20*	*2*		*200*	
Intermediate of Ta:	*50*	*2*	*200*	*500*	*One Month*
HCl	*conc.*	*2*		*1%*	
HNO_3	*conc.*	*4*		*2%*	

Working Standard # 1 is the 2% HNO_3 1% HCl instrument blank.

Working Standard # 2 is made by taking 1mL of Standard # 4 to 9mL of blank.

Working Standard # 3 is made by making a 1:1 dilution of Standard # 4 with blank.

NOTE: Solutions are to be prepared weekly.

ICV Intermediate:

(All from individual stock solutions, second source than calibration)

Compound/Standard:	*Initial conc.(mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
Au, Bi,	*1000*	*2*		*20*	
Ta	*1000*	*5*	*100*	*50*	
HCl	*conc.*	*10*		*5%*	*3 Months*
HNO_3	*conc.*	*20*		*10%*	

Working ICV

Compound/Standard:	*Initial conc. (mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
ICV (Intermediate): Au, Bi,	*20*	*1.1*		*110*	
Ta	*50*	*1.1*	*200*	*275*	*One Month*
HCl	*conc.*	*2*		*1%*	
HNO_3	*conc.*	*4*		*2%*	

CCV Intermediate:

(All from individual stock solutions, the same source as calibration)

Compound/Standard:	*Initial conc.(mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
Au, Bi,	*1000*	*2*		*20*	
Ta	*1000*	*5*	*100*	*50*	
HCl	*conc.*	*10*		*5%*	*3 Months*
HNO_3	*conc.*	*20*		*10%*	

Working CCV:

Compound/Standard:	*Initial conc.(mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
CCV (Intermediate): Au, Bi,	*20*	*1.1*		*110*	
Ta	*50*	*1.1*	*200*	*275*	*One Month*
HCl	*conc.*	*2*		*1%*	
HNO_3	*conc.*	*4*		*2%*	

Working Internal Standard Solution (Tb instead of Bi):

Compound/Standard:	*Initial conc.(mg/L):*	*mL used:*	*Total volume (mL):*	*Final Conc. (ug/L):*	*Holding Time*
Sc	*100*	*2.5*		*500*	
Y	*100*	*2.5*	*500*	*500*	*One Month*
Tb	*100*	*2.5*		*500*	
HNO_3	*conc.*	*10*		*2%*	

Annex 5

ICP-MS Quality Control Samples with Limits and Frequencies:

QC Type	Waters: Drinkwaters
Method	200.8
MS tuning solution 100ppb	Beginning of the run,5 replicates:RPD<5 Resolution at % peak heihgt:<0.75amu Mass calibration:<0.1 amu from TV
ICV	At start of every run Recovery range:90-110%
ICB	At start of every run not specified
CCV	Every 10 injections within 15% of TV
CCB	Every 10 injections <IDL
IS-sample	60-125% of IS observed in calibration std. (or dilute and re-run if is out)
MBLK	Every digestion batch of 20 <2.2 X MDL, < 10% sample
LCS	Every digestion batch within 15% of TV
MS	Every digestion batch within 30% of TV
MSD	Every digestion batch within 30% of TV, 20% RPD
Dilution Test	1/20 of each matrix. The sample is diluted 1+4 if it is >100IDL. RPD>10% indicates interferences.
PDS	As needed in 25% of TV, or interference present

QC Type	SW-846 (TCLP, Groundwater, solids)
Method	6020 A
MS tuning solution 100ppb	Beginning of the run,5 replicates:RPD<5 Resolution at % peak heihgt:<0.75amu Mass calibration:<0.1 amu from TV
Interference check A and AB	At the beginning of the run and every 12 hours
ICV	At start of every run Recovery range:90-110%
ICB	At start of every run <3 x IDL
CCV	Every 10 injections within 10% of TV
CCB	Every 10 injections </= 3 x IDL
IS-samples	>30% of IS observed in calibration blank (or dilute until %R>30%
MBLK	Every digestion batch <IDL
LCS	Every digestion batch of 20 within 20% of TV
MS	Every digestion batch of 20 within 25% of TV
MSD	Every digestion batch of 20 within 25% of TV, 20% RPD
Dilution Test	1/20 of each matrix. The sample is diluted 1+4 if it is >100IDL. RPD>10% indicates interferences.
PDS	As needed in 20% of TV, or interference present

TITLE: REPORTING DATA TO THE LIMS SYSTEM

KEY WORDS: Not applicable.

COMMENTS: The entire SOP is new.

PREPARED BY:	Laura Erickson	August 22, 2007

APPROVALS:

WET. LAB. GROUP LEADER:	Matt Gregory	August 22, 2007
QUALITY ASSURANCE MANAGER:	Mary Lubitov	August 22, 2007

REPORTING DATA TO THE LIMS SYSTEM

1.0 SCOPE AND APPLICATION

The SOP is used to determine the reporting procedure for OMEGA LIMS system.

2.0 TERMINOLOGY

Refer to EMT "Quality Manual" for definition of terms used in this SOP.

3.0 PROCEDURE

	To Report	SOP #
3.1	**3.1.1** The final ppm result will be calculated by the Omega LIMS system that will incorporate all dilutions (if applicable) and convert the data to a final result in mg/L (ppm). **3.1.2** To report the data to Omega, these steps are to be followed: From "Main screen", select "Data entry by run", select "add", Enter Instrument ID (Acidity/Alkalinity, KONELAB, BTU, HACH DR4000, MANUAL), Enter start date of analysis and the time, Enter analyst name that performed the test, Select "Load Samples", Select test code, Select samples analyzed from list, Select "View data", and enter the concentration results in the "Text Result" column. If any extra bench dilutions were made, be sure to include in the "DF" column on the front page of the data screens. **3.1.3** On the front screen, add any flags needed for the data in the "Comments" column. Sample specific flags must be put on the individual sample lines and batch specific flags must be entered on the LCS or MBLK lines which will paste into all the sample work order case narratives.	001, 002, 005, 007, 012, 014, 016, 027, 027A, 027B, 046
3.2	**3.2.1** MDL and PQL values in LIMS are based on 200mL aliquots used for analysis. Adjust MDL and PQL in LIMS if smaller aliquot is analyzed. **3.2.2** Samples needing a lower limit than < 0.40 mg/L will need to have a larger volume distilled.	004
3.3	Minimum reporting limit is established as 3 times MDL value. Samples should never be reported less than MDL. Adjust MDL and Reporting limit when an aliquot size is less than 200mL.	006
3.4	The reporting limit is based on MDL study based on 1g of sample used for the test. If less than 1g of sample were used, reporting limit should be adjusted.	007
3.5	**3.5.1** Both LCS results shall be entered into Omega. **3.5.2** After "read out" of samples is completed, the raw result prior to dilution should be reported into Omega (noted as "Result to report to LIMS") for samples, spikes, and duplicates with all corresponding total dilution amounts	008

		entered into the "DF" column on the first data table in Omega. All raw readings designated on the XL BOD sheets.	
	3.5.3	For the MS samples, the true value of the spike may need to be changed if there was a pre-dilution involved prior to the bottle dilutions. BOD sheet will give this value as "Spike true value with DF". Make sure that there is a reference to the original sample to get proper recovery calculation.	
	3.5.4	For duplicate samples, the raw values are put in with their first "QC" sample being set to "N" to avoid a duplicate result (values noted on sheet as Dilution # 1 and # 2 in "To LIMS" column). The reference to the original result should be provided to get RPD calculation.	
	3.5.5	If sample is exhibiting toxicity do not average the results. Report the result as > than the highest result. Insert comment "Sample is exhibiting toxicity".	
	3.5.6	The unseeded blank should be reported from the average of the four bottles.	
	3.5.7	Flagging: Flag the data associated with failed QC results or deviations from the standard conditions. The impact of deviations on the results should be explained. Here are some examples of such situations: BOD of dilution water > 0.2 mg/L; SCF < 0.6 mg/L or > 1.0 mg/L; DO loss in sample < 2 mg/L; DO final < 1mg/L; Temperature of incubator outside of 19-21°C; or Incubation time outside of range: 5 days +/- 5 hours.	
3.6		Flag samples associated with non-standard conditions or QC results out of control limits.	006, 009, 010, 040, 047, 068, 508, 524, 3600
3.7		Report results after calculation (Sec.11.1) upon standardization of mercuric nitrate (Sec.9.0).	011
3.8		Flag the results associated with failed MS/MSD, RPD or any deviation from the SOP.	004, 015, 016, 019, 029, 032, 033, 034, 036, 039, 044, 049, 050, 052, 053, 054, 055, 058, 059, 060, 062, 063, 064, 069, 072, 073, 074, 075, 076, 077, 078, 079, 080, 081, 082, 083, 084, 086, 087, 089, 090, 091, 093, 101, 109, 117
3.9	3.9.1	Minimum reporting limit is established as 3 times MDL level. Samples should never be reported less than MDL. Adjustment of reporting limit might be needed if less than 50 mLs of liquid sample and less than 1g of solid sample was used for distillation; or when less than 20mL of distillate was used for colorimetric reading.	016
	3.9.2	Save electronic data produced by HACH DR 4000 in the directory: Data1 on Ralph (L:)\Wc\HACH. Document in the lab, book data file name.	
3.10		Report fluoride concentration, which was determined from the pH meter if it is within calibration range.	024
3.11	3.11.1	Reporting limit (PQL) should be at or above the lowest	029

	calibration standard. Samples exceeding the highest standard should be diluted and reanalyzed. **3.11.2** Report the instrument reading and the dilution factor to OMEGA. Omega will calculate the final result and adjust the reporting limit. **3.11.3** Report anions NO_2 and NO_3 as N; and PO_4 as P. **3.11.4** Special Reporting Requirements for Chlorine, Sulfur and Bromine prepared by combustion. The bomb preparation test code is 5050_PR. MDL values for all matrices are based on initial sample size = 1g and final volume of washings = 100mL. Dilution factor 5 is incorporated into MDLs by default. The MDL and PQL have to be adjusted manually if the dilution factor > 5. The conversion factors used for the analytical test codes are: %S = 0.00000033 (3.3E-8); %CL & %Br-= 0.0000001(1E-7); IC_300_Eproduct (Cl and Br) = 0.001. Conversion factors eliminate manual calculations and convert the IC reading "mg/L" into final concentration –"%" or "mg/kg". Report to OMEGA prep information: initial sample wt (kg) and total volume of washings (mL). For MS/MSD, enter weight of spike added to sample. Do not enter the wt of the sample. Report to OMEGA IC reading multiplied by dilution factor. Subtract the method blank from sample for chlorine only. Adjust "Raw Spike" value in OMEGA, because spike varies with the weight of standard added to prepare LCS or MS/MSD. To calculate "Raw Spike" multiply TV of standard by gram of standard used. Reporting IC data for MS/MSD, subtract sample value from MS value. Make sure that there is no spike reference for MS or MSD. In order for OMEGA to calculate RPD correctly, equal amount of spike should be added to MS and MSD. **3.11.4.1** Example 1 (LCS): Mineral oil standard contains 0.336% of Chlorine. 1.1g of standard was combusted. Report 0.0011kg as initial wt. Raw spike value for OMEGA is 0.3360 x 1.1 = 0.3696%. **3.11.4.2** Example 2 (MS/MSD): 0.2g of sample was spiked with 0.6g of Mineral oil standard containing 0.336% Chlorine. Report 0.0006kg as initial wt for MS. Raw spike value for OMEGA is 0.3360 x 0.6 = 0.2016%.	
3.12	Report only values that fall between the lowest and the highest calibration standards.	032
3.13	**3.13.1** The MDL and PQL in OMEGA are based on aliquot size 250mL or g for liquids and for soils. **3.13.2** The MDL and PQL adjustment per aliquot size and final volume of distillate is done by the OMEGA. Refer to SOP # 261.	036
3.14	**3.14.1** Report the first of the duplicate results. **3.14.2** Report the pH of the calibration verification buffers	038

<table>
<tr><td></td><td>analyzed with the batch.

3.14.3 Flag the data associated with any and non-standard situations.

3.14.4 If samples are very caustic or very acidic and the measured pH is outside of bracketing calibration buffers (< 1 or > 12.45), report the measured value and flag the data.</td><td></td></tr>
<tr><td>3.15</td><td>3.15.1 PQL should be adjusted in OMEGA when aliquot size is less than 300mL.

3.15.2 Samples associated with failed QC should be flagged when reported.</td><td>044, 090</td></tr>
<tr><td>3.16</td><td>Report, "PASS" if it is reached the highest temperature.</td><td>051</td></tr>
<tr><td>3.17</td><td>3.17.1 The reporting limit of 2 mg/L is approximately 3 times the MDL. When less than 50mL of sample is used for titration the reporting limit should be adjusted.

3.17.2 Flag the data if sample was in the container with the headspace.</td><td>057</td></tr>
<tr><td>3.18</td><td>Report turbidity readings in NTU as follows:

<table><tr><th>Turbidity range</th><th>Report to nearest</th></tr><tr><td>0 – 1</td><td>0.05</td></tr><tr><td>1 - 10</td><td>0.10</td></tr><tr><td>10 - 40</td><td>1.00</td></tr><tr><td>40 - 100</td><td>5.00</td></tr><tr><td>100 - 400</td><td>10.00</td></tr><tr><td>400 - 1000</td><td>50.00</td></tr><tr><td>> 1000</td><td>100.00</td></tr></table></td><td>064</td></tr>
<tr><td>3.19</td><td>3.19.1 The pH of extract, date of extraction (start and finish), and analyst that extracted sample all needs to be entered into Omega computer system after completion (which is to include the MBLK if it was extracted with the batch).

3.19.2 Anything out of the ordinary may be entered as a comment to "flag" data about any problems that may have occurred during the sample preparation.</td><td>070, 085</td></tr>
<tr><td>3.20</td><td>Report each sample in duplicates.</td><td>073</td></tr>
<tr><td>3.21</td><td>3.21.1 Preparation Data: enter the initial weight (kg) of sample and the final volume of washings (mL).

3.21.2 Analytical Data: multiply the result (mg/L) determined by IC by dilution factor, subtract method blank then report calculated value to OMEGA.

3.21.3 Raw value of spike needs to be adjusted for OMEGA, because the value varies depending on the weight of standard used to prepare LCS or MS/MSD. To calculate the raw spike value, multiply the target value by the weight of standard used for LCS or MS/MSD preparation.</td><td>088</td></tr>
</table>

	3.21.3.1 Example A: Standard -Mineral oil contains 0.336% of Chlorine. 1.1g of standard was combusted. Adjusted raw spike value = 0.3360 x 1.1 = 0.3696%. **3.21.3.2** Example B: 0.2g of sample was spiked with 0.6g of standard mineral oil containing 0.336% Chlorine. Adjusted raw spike value = 0.3360 x 0.6 = 0.2016%. **3.21.4** In order for OMEGA to calculate RPD correctly, add equal amount of mineral oil standard to matrix spike and matrix spike duplicate. **3.21.5** Flag the data associated with any non-standard conditions that could affect the final result.	
3.22	Enter the percentage of organic matter on a wet weight basis as determined in section 11.1 in SOP # 93 into Omega.	093
3.23	Initial volume (mL), final volume (mL), date digested (start and finish), and analyst that digested sample all needs to be entered into Omega computer system after completion (which is to include the MBLK, LCS, MS, and MSD samples and volumes). Labels can be generated after this information is entered into Omega. The MBLK and LCS samples should have 40mL initial volume and final volume of 50mL for correct calculation during an upload of data. Anything out of the ordinary may be entered as a comment to "flag" data about any problems that may have occurred during the sample preparation.	101
3.24	**3.24.1** Report to the OMEGA all quality control samples: ICV, ICB, CCB, CCV, LCS, MB, LL check, MS/MSD. **3.24.2** Double check entered results to make sure the numbers are reported correctly. **3.24.3** Samples with raised reporting limits due to the interferences or QC problems should be flagged with a note explaining the problem. The corresponding note should be provided on the raw data. Questionable results on regular and/or QC samples must be discussed with the supervisor or QA manager for plan of action.	102, 103
3.25	**3.25.1** Initial weight (Kg), final volume (mL), date digested (start and finish), and analyst that digested samples all needs to be entered into Omega computer system after completion (which is to include the MBLK, LCS, LCS1, MS, and MSD samples and volumes). Labels can be generated after this information is entered into Omega. The MBLK and LCS samples should have 0.001 initial weight and final volume of 100 mLs for correct calculation during an upload of data. **3.25.2** Anything out of the ordinary may be entered as a comment to "flag" data about any problems that may have occurred during the sample preparation.	104
3.26	**3.26.1** Raw results should be reported from instrument data into the raw data column in Omega. The digestion dilutions will be put in automatically from the prep entries in Omega, but any bench dilutions will need to be entered	109, 111

	into the "DF" column in Omega. **3.26.2** Samples run by MSA or with results raised due to interferences or QC problems should have a note indicating the problem in the comments column. A separate note on the raw data should also be provided. **3.26.3** Questionable results or QC on samples should be consulted with supervisor or director for plan of action or flagging before reporting results. **3.26.4** All needed documentation of problems, sample dilutions, sample initial and final volumes, standards traceability, spike traceability and values, date, and analysts' initials should all be on the actual raw instrumental data. **3.26.5** Any corrections made on raw data should be crossed out with a single line with the date and analysts' initials. Re-runs should be documented with the location of the re-run sample (date or instrument specification. **3.26.6** All sample types should be clearly marked in run, refer to abbreviation list for each analyst and instrument for term definitions that is included with every run.	
3.27	Initial weight, final volume, date and time digested (of extraction), the preparation analyst name all need to be entered into LIMS. Anything out of the ordinary, problems occurred during the sample digestion should be entered as a comment and used to "flag" the data.	116, 8318, 3510, 3540, 3550
3.28	**3.28.1** Enter in the OMEGA date and time of preparation and the name of preparation analyst, initial volume and final volume of sample. **3.28.2** Anything out of the ordinary should be entered in a comment column to alert about problems that may have an impact on the quality of the data.	117
3.29	**3.29.1** "Flagging" refers to the addition of a note into the computer with the final result that will go to the client to inform them of any problems there may be with the results. **3.29.2** Raw results should be reported from instrument data into the raw data column in Omega. The digestion dilutions will be put in automatically from the prep entries in Omega. Additional bench dilutions need to be entered into the "DF" column in Omega. Reporting limits are generally 3 times the MDL for either instrument to keep reported values consistent and ensure that client's MCL's are met. MDLs and PQLs (reporting limits) will be adjusted in Omega by digestion and bench dilutions. Limits attached with MDL lists at end of SOP. **3.29.3** Samples run by MSA or with results raised due to interferences or QC problems should also be flagged in the computer with a note indicating the problem. The note can be put in the comments column in Omega. A separate note on the raw data should also be included.	118, 119

	Questionable results or QC on samples should be consulted with supervisor or director for plan of action or flagging before reporting results. **3.29.4** Analytes above calibration range but within 90% of Linear Dynamic Range need to be flagged in the comments.	
3.30	**3.30.1** All needed documentation of problems, sample dilutions, sample initial and final volumes, spike values, date, and analysts' initials should all be on the actual raw instrumental data. **3.30.2** Any corrections made on raw data should be crossed out with a single line with the date and analysts' initials. **3.30.3** Re-runs should be documented with the location of the re-run sample (date or instrument specification). **3.30.4** All sample types should be clearly marked in run, refer to abbreviation list for each analyst and instrument for term definitions that is included with every run.	119
3.31	**3.31.1** Initial volume (mL), final volume (mL), date of extraction (start and finish), and analyst that extracted sample all needs to be entered into Omega computer system after completion **3.31.2** The MBLK and LCS samples should have 100mL initial volume and final volume of 0.5mL for correct calculation during an upload of data. **3.31.3** Anything out of the ordinary may be entered as a comment to "flag" data about any problems that may have occurred during the sample preparation.	548
3.32	**3.32.1** When analysis is complete, download the final sample results into the OMEGA. If a sample has been diluted then make sure that the dilution factor is entered correctly. Make sure that each sample has correct reference to the method blank, and CCV. Add the appropriate qualifiers and comments to the sample data after performing a complete review of the sample data and QC data. **3.32.2** The results for Chlordane based on the peak average should be entered manually. **3.32.3** The reported data is reviewed and authorized in the OMEGA by a supervisor. **3.32.4** The SOP includes contingencies for data associated with non-compliant QC results. This kind of data can be reported only with permission of supervisor. The impact of QC results on the data has to be clearly explained in the comment section.	8081/608/508
3.33	When analysis is complete, download the final sample results into the OMEGA. If a sample has been diluted then make sure that the dilution factor is entered correctly. Make sure that each sample has correct	8270/625

	reference to the method blank, and CCV. Add the appropriate qualifiers and comments to the sample data after performing a complete review of the sample data and QC data. The reported data is reviewed and authorized in the OMEGA by a supervisor.	

4.0 REFERENCES

4.1 For list of SOPs refer to table in Procedure, Sec.3.0.

4.2 Laboratory Quality Assurance Manual, Environmental Monitoring & Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.

TITLE: CONTROL CHARTS

KEY WORDS: Statistical Quality Control Charts or SQC charts

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY: Brian Goyette February 11, 2008

APPROVALS:

QUALITY ASSURANCE MANAGER: Lubitov February 11, 2008
Mary Lubitov

QUALITY ASSURANCE MANAGER: M Goyette February 11, 2008
Brian Goyette

CONTROL CHARTS

1.0 SCOPE AND APPLICATION

1.1 Statistical Quality Control (SQC) charts are used in the laboratory to monitor the stability of the measurement process. They are an excellent tool in assuring the analyst, laboratory management, or any outside accrediting authority that the analytical system remains in control or is stable. Changes in recoveries or trends are indicative of problems in the analytical system, i.e. instrument instability or degraded standards, and should be addressed by the analyst. Significant changes should initiate the corrective action process. Corrective action could include, prepping new daily standards, recalibrating, or instrument maintenance etc. There are two types of control charts used in the laboratory, the x bar chart and the R chart.

1.2 The x-bar chart is a plot, in time order, of the recoveries of replicate measurements of a specific analyte as compared to the calculated average of a set of replicates of the same analyte and same matrix, or method specified limit. X-bar charts are a means to assess the accuracy of the analytical system. Five lines are placed on the chart to aid in interpretation, a center line which is typically an average recovery, upper and lower warning limits $\pm 2\sigma$, or $\pm$ 2 times the standard deviation, and upper and lower control limits which are $\pm 3\sigma$. X – bar control charts are used to monitor the recoveries of matrix spikes, matrix spike duplicates, laboratory control samples, matrix duplicates, and surrogates in specific sample matricies. The results can be compared to method specific acceptance criteria or where no method specific limits are provided used to calculate in house limits. In house limits are updated and reviewed approximately every 6 months. Significant changes in recovery limits will initiate the corrective action process. Where method specific acceptance criteria is not provided and there are not enough data points to create in house limits default values will be used until enough data points have been collected to develop in house limits.

1.3 The next type of control chart is an R chart or range chart. This type of control chart monitors analytical precision between duplicate measurements of the same method or analyte in the same sample matrix. The R control chart consists of a center line which is the average relative percent difference of a series of measurements or a method specified RPD limit. R control charts are used to monitor the relative percent differences between matrix spikes/matrix spike duplicates, matrix duplicates and other duplicate measurements of the same analyte in the same sample matrix. The results can be compared to method specific acceptance criteria or where no method specific limits are provided used to calculate in house limits. In house limits are updated and reviewed approximately every 6 months. Significant changes over time of the relative percent differences between measurements will initiate the corrective action process. Where method specific acceptance criteria is not provided and there are not enough data points to create in house limits default values will be used until enough data points have been collected to develop in house limits.

1.4 *Control charts, for specific analytes, are generated and reviewed monthly by the QA Manager. These charts are reviewed, printed out, and stored in a binder in the QA office. Review consists of verifying that all recovery outliers are flagged in the LIMS. If outliers are not flagged, the supervisor will be notified. Consistently missed or un-flagged outliers will initiate/require a formal corrective action investigation. If 7 of more consecutive data points are found on either side of the mean, the supervisor will be notified and the analytical system will be inspected. Corrective action may include; recalibrating, re-preping standards, or spike solutions.*

1.5 *Supervisors will also be notified if trends indicate potential problems with the analytical system.*

1.6 *Situations which may not require supervisor notification include situations where the recovery windows are very narrow either due to method required windows, 90% - 110%, or internally generated windows such as those for Total Dissolved Solids with limits of 94% -103%. In situations such as these if 7 data points are found above or below the calculated mean, but are near perfect recoveries, near 100%, no action will be taken.*

2.0 TERMINOLOGY

2.1 Average or the mean of X, $\overline{x}$, is defined as:

$$\overline{x} = \frac{1}{N}\sum_{i=1}^{N} x_i = \frac{x_1 + x_2 + \cdots + x_N}{N}$$

Where:
Xi = individual measurement
N = number of measurements

2.2 Standard Deviation or σ =

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(x_i - \overline{x})^2}$$

2.3 Relative Percent Difference or RPD =

RPD = **[X1 - X2} / Xave x 100**

3.0 PROCEDURE

3.1 To construct an x-bar chart collect a minimum of 7 measurements, preferably 15-20, done on different days. Use the equations above to calculate the average or mean recovery and the standard deviation. Use the calculated average recovery and standard deviation to create the x-bar control chart limits. Where:

Centerline	$\overline{x}$ ave or known value
UWL (Upper Warning limits)	$\overline{x} + 2\sigma$
UCL (Upper control limits)	$\overline{x} + 3\sigma$
LWL (Lower warning limits)	$\overline{x} - 2\sigma$
LCL (Lower control limits)	$\overline{x} - 3\sigma$

3.2 All recovery outliers shall be flagged in the LIMS with an S flag and comments added to the case narrative identifying all recovery outliers. Tests or specific analytes that consistently fail or have recoveries that exceed limits indicate a problem with the analytical system. When such trends are noted inspect the analytical system to determine the source of the problem. Potential areas of concern are, degraded standards, loss of instrument calibration, instrument drift, or other problems determined by the analyst. All action taken needs to be documented in the appropriate place, such as daily run log, instrument maintenance log, standard prep log book etc. When significant problems are determined, the QA group should be notified and the formal corrective action process initiated.

3.3 To construct an R chart collect approximately 20 data points, preferably a larger set, analyzed on different days. Calculate the relative percent difference (RPD) of the concentrations between matrix duplicates, matrix spike/matrix spike duplicate, or laboratory control sample and its duplicate. From this group of RPD' s calculate an average RPD. The average RPD value obtained from the data points is entered into the LIMS and used as the control limit or centerline on the control chart. All RPD outliers shall be flagged in the LIMS with an R flag and comments added to the case narrative identifying all RPD outliers.

3.4 Tests or specific analytes, which consistently fail or have RPD's that exceed limits, indicate a problem with the analytical system. When such trends are noted inspect the analytical system to determine the source of the problem. Potential areas of concern are, inconsistent sample delivery techniques, instrument problems causing erratic response, poor sample prep techniques or other problems determined by the analyst. All action taken needs to be documented in the appropriate place, such as daily run log, instrument maintenance log, standard prep log book etc. When significant problems are determined, the QA group should be notified and the formal corrective action process initiated.

3.5 The Grubbs test may be used as a statistical tool to determine if a single data point, percent recovery or RPD value is an outlier relative to the rest of the data in the data set, and may be rejected. The Grubbs test detects one outlier at a time. This calculation is part of the omega LIMS.

4.0 REFERENCES

4.1 ISO/IEC 17025,General requirements for the competence of testing and calibration laboratories. ISO/IEC 17025:1999(E)

4.2 Laboratory Quality Assurance Manual, Revision 10.0, Effective date 5/1/2006. Environmental Monitoring & Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.

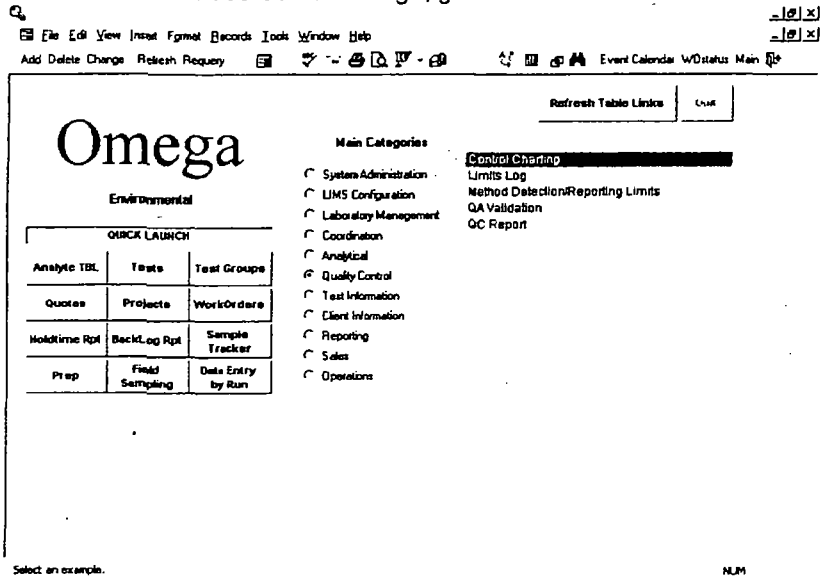
4.3 NELAC, National Environmental Laboratory Accreditation Conference, Quality Systems Chapter 5, Appendix D. 6/05/2003

4.4 USEPA, OSWER. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. Third Edition (November 1986), Final Update III: Method 8000 B, Rev. 2, (12/96), Determinative Chromatographic Separations.

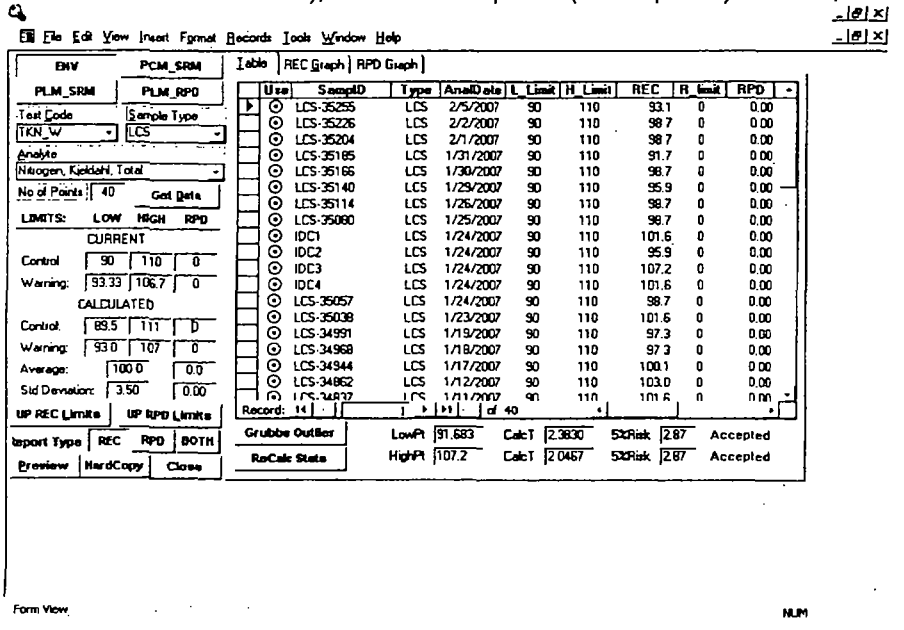
APPENDIX A

Control charts can be used to check for recovery trends in the laboratory. Trends can be either a gradual increase or decrease of recovery data and are indicative of a problem with the analytical system. A single spike in recovery is typically an isolated event. Control charts can be used to monitor: CCVs, LCSs, MSs, and surrogate recoveries. R or range control charts are used to compare the differences between duplicate measurements such as, MS/MSDs, sample duplicates, LCS/LCD, etc.

1. From the front screen of Omega, get to control charts from "Control Charting".



2. Select test code, sample type (LCS, MS, or MSD for RPD), select analyte (only one shown if no selection list exists for test), for number of points (No. of points) enter 30, and click "Get Data".



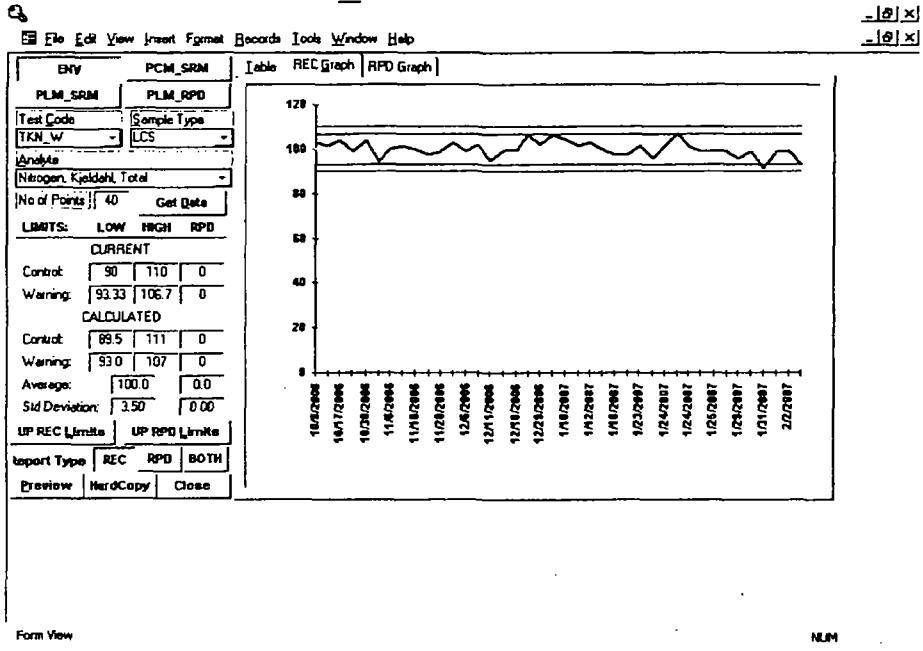
3. From this TKN water example, the Control limits would not be updateable as the Calculated limits are greater than the Current limits. If Calculated were smaller, the limits could be updated.

4. If there was one particular recovery that appeared to be very outside of the average, the Grubbs test can be used to determine if it can be removed from the data set.

5. For an LCS sample, to check for trends, select "REC Graph" and look to see if 7 or more points are consistently or consecutively on one side of the central average value, if so that indicates a trend.

6. If a trend is occurring, print out the graph, take corrective action and notify your supervisor.

7. The graph shown in this example is an x-bar or standard recovery control chart, which consists of the 5 lines as mentioned in section 1.2. The center line which in this example is 100%, the center line can also be calculated from the average of several data points, the next closest lines to the center dotted line are the warning limits which are calculated as ± 2 standard deviations, and the outer most lines are the action or control limits which are ± 3 standard deviations.



TITLE: QUALITY CONTROL

KEY WORDS: Not applicable.

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY:	Brian Goyette	April 11, 2008

APPROVALS:

QUALITY ASSURANCE MANAGER:	Mary Lubitov	April 11, 2008
QUALITY ASSURANCE MANAGER:	Brian Goyette	April 11, 2008

QUALITY CONTROL

1.0 SCOPE AND APPLICATION

1.1 *The quality control procedures incorporated in the laboratory preparation and analytical procedures insure that these protocols, as specified in the EMT Quality Assurance Manual (QAM) are followed. Some examples are presented in this document.*

1.2 *All quality control measures are assessed and evaluated on an ongoing basis. Recovery limits are generated either internally or method required limits are used. The laboratory LIMS is used to generate internal recovery limits. Matrix specific percent recovery (%R) and relative percent difference (RPD) are generated when a minimum of 30 data points are available. For specific examples on the use of Statistical Quality Control Charts please refer to SOP #213 Control Charts.*

1.3 *Procedures exist to address situations where quality control samples have recoveries, which exceed the recovery limits. This includes re-analysis, re-preparing the QC and all associated samples, and flagging associated data. For specific procedures please refer to the specific method or the EMT Quality Assurance Manual.*

1.4 *In addition to the internal QC samples mentioned in this document EMT participates in several double blind Performance Testing studies that, cover most laboratory matrices and methods. For further details of the EMT PT program refer to the Quality Assurance Manual.*

2.0 TERMINOLOGY

Refer to the EMT "Quality Assurance Manual" for terms used in this SOP.

3.0 PROCEDURE

3.1 Blanks

3.1.1 *The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank is processed along with, and under the same conditions, as the associated samples to include all steps of the analytical procedure. Blanks can be used to determine if any of the reagents used are contaminated. The method blank is analyzed before any of the associated samples are analyzed. If the blank is found to contain target analytes or, any type of matrix contamination, re-analyze the blank to verify. Any samples associated with a contaminated method blank are either re-processed, re-prepared, or the results reported with appropriate flags.*

3.1.2 *Some methods require that any* blank readings be subtracted from the readings obtained for the samples or used to set the zero point depending on the test.

3.1.3 At least one blank must be *prepared and analyzed* per batch, *for all analysis. The method blank must consist of the same quality system matrix as the associated samples and must be shown to be free of the analytes of interest.* A high response in a blank indicates a problem *and corrective action must be taken.*

3.2 *Matrix* Duplicates

3.2.1 *Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.*

3.2.2 *Matrix* duplicates should be run every 10 samples *or, at the frequency required by the method.* If *an analytical* batch is less than 10 samples, run at least one duplicate. *The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as the Relative Percent Difference or RPD. The calculated RPD must be within the limits required by the method, or the limits established by the laboratory.*

3.2.3 *The RPD* is calculated by the following *equation:*

$$RPD = ((D1 - D2) / Ave.) \times 100$$

3.3 ***Matrix* Spikes**

3.3.1 *Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. A matrix spike and matrix spike duplicate are prepared and analyzed with every 20 samples, or more frequently if required by the specific method.*

3.3.2 *The analytes spiked should be either those specified by the test method or those target analytes most common to the test procedure for multi analyte procedures. The concentration of the spiked analytes should be close to the mid point of the method calibration.*

3.3.3 *The percent recovery of the spiked analytes should be within the limits either specified by the test method or those determined internally.*

3.2.3 The %Recovery for the Spike is calculated by the following *equation:*

$$\% \text{ Recovery} = ((\text{Spiked Sample} - \text{Sample}) / \text{Spike}) \times 100$$

3.3.4 *The results from the matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), and relative percent difference (RPD).*

3.4 ***Laboratory Control Samples***

3.4.1 *Laboratory Control Samples (LCS's) are used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is "out of control". Any samples associated with an out of control LCS are reprocessed for re-analysis or the results reported with the appropriate data qualifying flags.*

3.2.3 *The %Recovery for LCS* is calculated by the following *equation:*

$$\% \text{ Recovery} = ((\text{Known Value} - \text{Analytical Value}) / \text{Known Value}) \times 100$$

3.4.2 *Typically a LCS is prepared at a minimum of one per preparation batch for most analysis. However, some analysis does not have spiking solutions so LCS's are not required, some examples include pH, solids, temperature etc.*

3.5 ***Performance Testing***

3.5.1 *EMT participates in the following Proficiency Testing(PT) studies, at the required frequency, to maintain NELAC accreditation for Drinking Water, Hazardous and Solid Waste, and Waste Water: January and July Water Supply (WS), April and October Water Pollution (WP), April and October Soil/Hazardous Waste, June DMR-QA.*

3.5.2 *All formal PT studies are obtained through approved PTOB/PTPA PT providers. For continuing accreditation, completion dates of successive proficiency rounds for a given PT*

field of testing shall be approximately six months apart, with the successful completion of two acceptable PT studies for each field of testing out of the most recent three. Failure to meet this semiannual schedule is regarded as a failed study.

3.5.3 *For further information on PT studies refer to the EMT Quality Assurance Manual.*

3.6 *Miscellaneous Quality Control Standards*

3.6.1 *In addition to the QC samples mentioned, some methods require an Initial Calibration Verification (ICV) standard. This is a laboratory prepared standard, it is from a source, which is different from the calibration standards. This standard is run as a secondary source after a calibration to verify the correctness of the calibration. The recovery limits for these standards are the same as those for the Continuing Calibration verification (CCV) standard.*

3.6.2 *Surrogates, common to organic methods, are a means to monitor to entire analytical process. Surrogate compounds are similar to the target analytes. They are added to the sample prior to extraction, and go through the entire prep process. Surrogate recoveries are either generated internally or are specified by the method. For further information on surrogates please refer to the specific methods procedure.*

3.6.3 *Other QC samples include Post Digestion Spikes, used for some metals analysis to verify matrix issues.*

4.0 REFERENCE

Laboratory Quality Assurance Manual, Environmental Monitoring and Technologies, Inc., 8100 North. Austin Avenue, Morton Grove, IL 60053-3203.

Environmental Sample Receipt and Handling

1.0 Scope

1.1 The SOP covers sample receipt by the laboratory, login procedures, and sample preservation and storage. This covers all sample types except those considered e-products.

2.0 Summary

2.1 This SOP applies to every EMT personnel that take possession of a sample on behalf of the laboratory. Samples can be shipped, usually in an insulated ice cooler, delivered by EMT field personnel or directly delivered by the client.

2.2 Review of the COC (chain of custody), reconciling sample label information against the COC, documentation of discrepancies or problems with samples observed upon receipt, login of the sample into the LIMS (laboratory information management system) and assigning of unique laboratory number are discussed.

2.3 Samples are also checked that proper preservation and handling occurred during transfer to the laboratory.

3.0 Apparatus

3.1 Thermometer, such as the Infrared Thermometer "Baxter" Model T 2940. The accuracy of the thermometer is verified monthly against a NIST traceable thermometer.

4.0 Reagents and Materials

4.1 Sulfuric acid (1:1) H_2SO_4. ACS grade.
4.2 Hydrochloric acid (1:1) HCL. ACS grade.
4.3 Nitric acid (1:1) HNO_3. "For trace Metal Analysis" grade.
4.4 Sodium Hydroxide (NaOH) pellets. ACS grade reagent
4.5 Sodium thiosulfate ($Na_2S_2O_3$) crystals. ACS grade reagent
4.6 pH paper
4.7 Zinc Acetate solution (2N), Zn $(CH_3COO)_2$ *2 H_2O. Dissolve 110 g zinc acetate dihydrate in 500 ml of reagent water.
4.8 Disposable transfer pipettes, 2 ml.
4.9 Stainless steel bowl
4.10 Stainless steel spoon
4.11 Color tapes or color-coded preservation labels

5.0 Safety Hazards

5.1 Environmental samples must always be considered to be potentially hazardous to the health. The samples may have toxic, corrosive, explosive, and flammable properties. The minimum protection consists of eye protection, latex gloves, and lab coat.

6.0 Procedure

6.1 **Sample Receipt:** The Sample Login Technician or equivalent EMT employee receives samples at the laboratory. The samples are usually shipped in an insulated ice cooler with the bottles stabilized in the container and covered with ice, or collected and delivered by EMT personnel.

6.2 **Temperature measurement:**

6.2.1 Temperature measurement is not required when the samples are received the day they were collected in a cooler still containing bits of not melted solid ice. ("Blue ice" packages are not considered to be a solid ice). Mark on the COC whether the samples are received on ice.

6.2.2 Take samples out of the chest and measure the temperature of each container type (plastic or glass); per work order; per cooler using IR thermometer T2940:

- Aim the sensor of the instrument at the sample, pull and hold the trigger. The distance between the sensor and the sample should be approximately 10-15 cm.
- The temperature and emissivity will be displayed. Adjust emissivity if needed by pressing the + or - buttons. Make corrections to the measured temperature value according to the last instrument verification. Document the temperature of the sample in the COC.
- To stop measuring, release the trigger. The last value can be displayed at any time by pressing RECALL. Each pull of the trigger erases the previous reading and starts a new measurement.
- Record the result of measurement on COC.

All samples that require thermal preservation are considered acceptable if the arrival temperature is ranging from just above freezing temperature of water to 6° C.
If the temperature requirements are not met, print out from OMEGA (LIMS) the checklist with the marked discrepancy, and record the temperature. Submit the checklist to the login supervisor for further actions.

6.3 **Sample Identity:** The information needed to identify the samples is contained in the chain of custody (COC) and on the label of each sample container. The COC should be attached to the lid or inside of the cooler. This form lists a client name, description of the sample, who collected it, in what container, and where and when the sample was collected. The COC includes the requested test parameters, preservatives used and comments on the condition of sample. The field labels attached to samples contain the name of the client, date, time, exact location of sampling and sample description. ***The information on the field label must agree with the chain of custody entry.*** The person delivering the sample should write down the date and time of delivery and sign the COC as relinquished. The person receiving the sample should also mark down the date and the time of receipt and sign the COC.

6.4 **Sample Integrity** is verified by visual inspection. Any problem noted (broken, cracked or inappropriate container, loose cap, air bubbles in samples for volatile compounds, incorrect chemical preservative, not sufficient sample volume etc.), must be entered in the checklist in Omega. The checklist with noted deficiencies is printed out and passed on to the login supervisor together with COC. See Appendix 1 for appropriate containers and chemical preservatives.

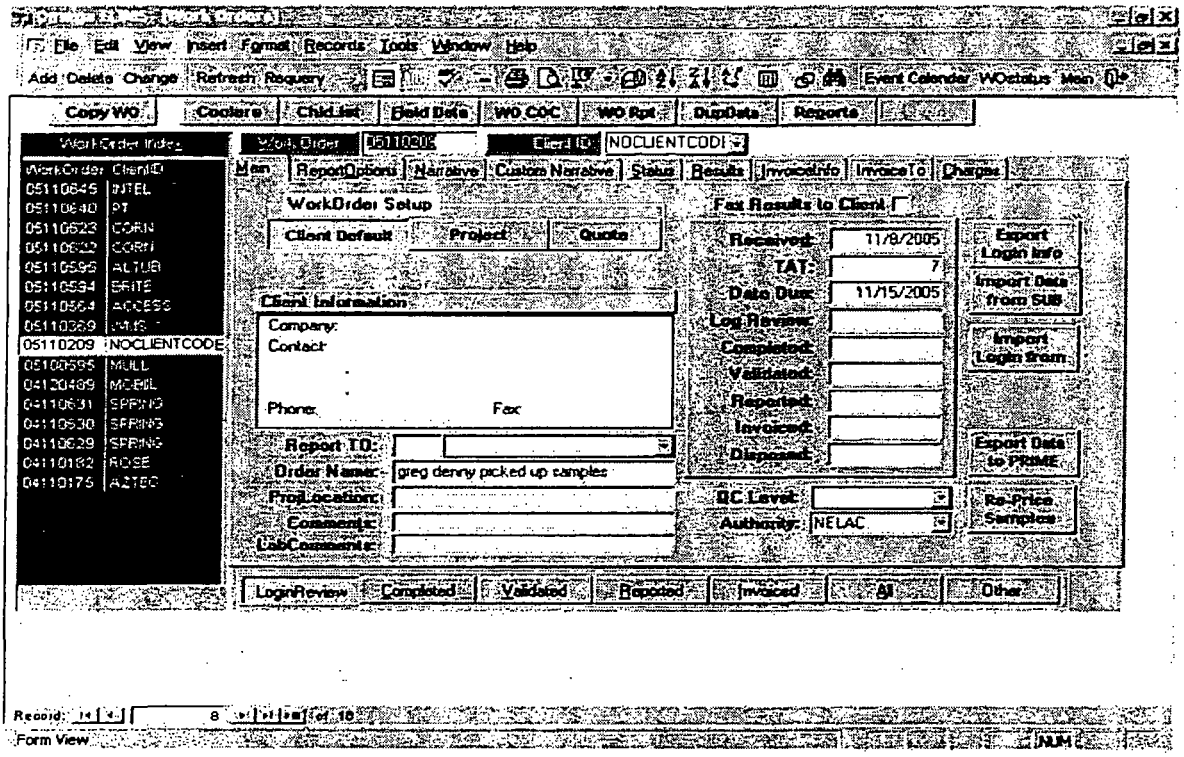
6.5 ***Omega II Login Procedures:*** Upon receipt, sample information is transferred from the COC and field label to the central computerized Laboratory Information Management System (LIMS) as follows:

6.5.1 Once in the Omega II system click arrow on work orders. The screen will appear as below. (Fig. 1) Click on **Add** located at the upper left side of the screen. A new work order number will appear.

6.5.2 Go to **Client ID** and click on the arrow in the box next to it. A list of client names and codes will appear, select the proper client and click twice. The information for that client will now be in the **Client Information** box.

6.5.3 If on the Chain of Custody (COC) it is noted **Project** or **Quote** the sales representative has previously set up the tests for that client. Each project or quote has an ID and a project name that will also be on the COC. By clicking on the appropriate box (Project or Quote) a list of possible ID's appear (Fig. 2). Choose the ID that is written on the COC in order for the preset project or quote to be activated for the login session. If the project or quote is not written on the COC double click on the **Client Default** box instead. Ex: If a "project ID" is written on the COC click on project, a list of project ID's and project names will appear. Choose the corresponding ID and click twice. If a "quote ID" is written on the COC click on quote, a list of quote ID's and project names will appear. Choose the correct ID and click twice.

Fig. 1



6.5.4 Before any samples can be logged in the date received must be recorded in the box next to **Received**. The **Date Due** will automatically appear for normal turn around time (TAT) unless it is manually changed in the **TAT** box (Fig. 1).

6.5.5 Note any general lab comment that may be on the COC in the **Lab Comment** field (see fig 1)

6.5.6 Once the date has been entered and the TAT verified click on the box that says **LOGIN** (In red). You will now be in the Login screen (Fig. 3)

Fig. 2

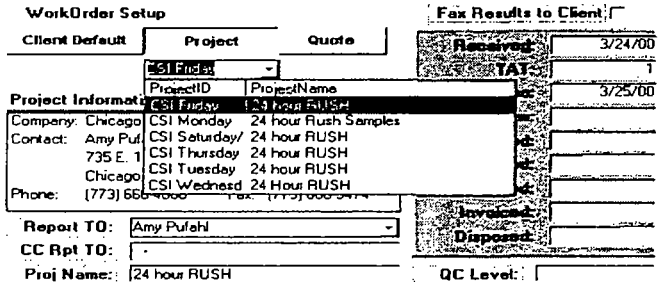
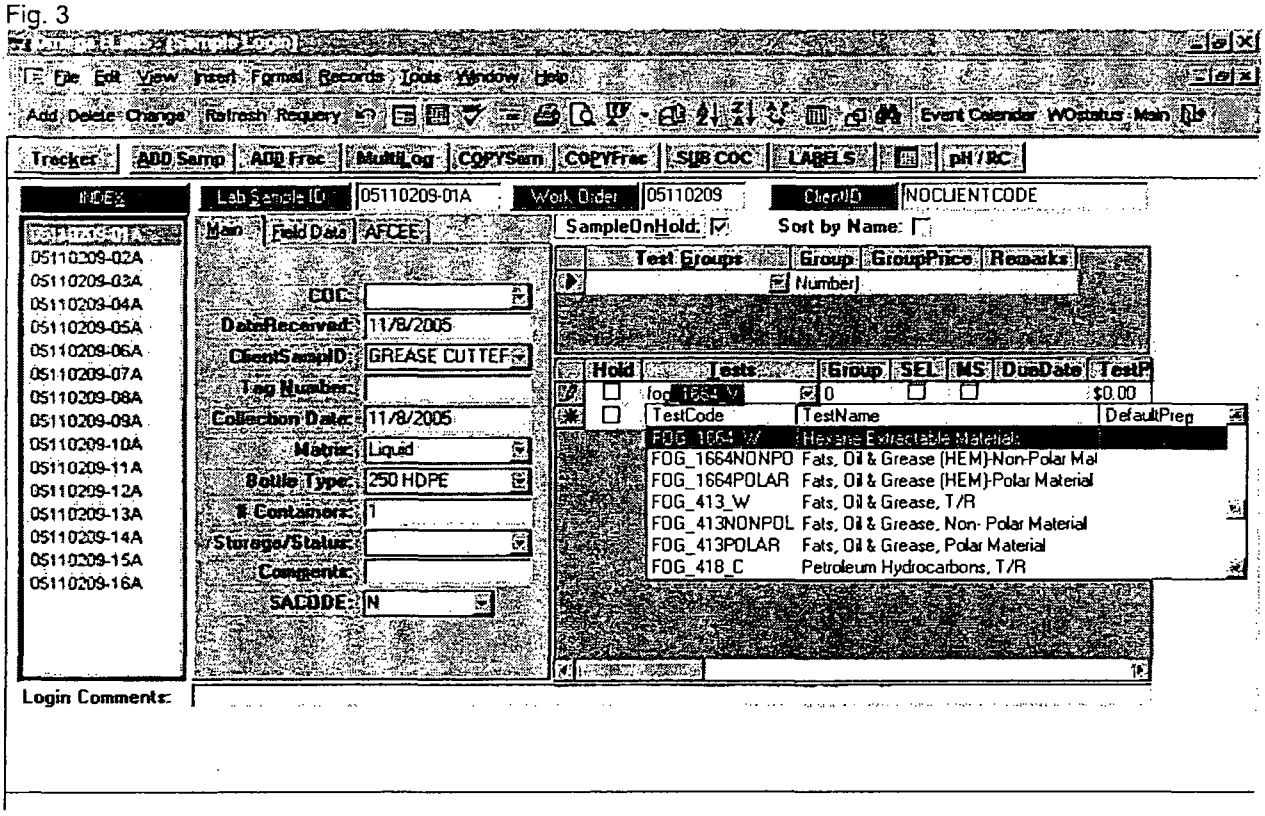
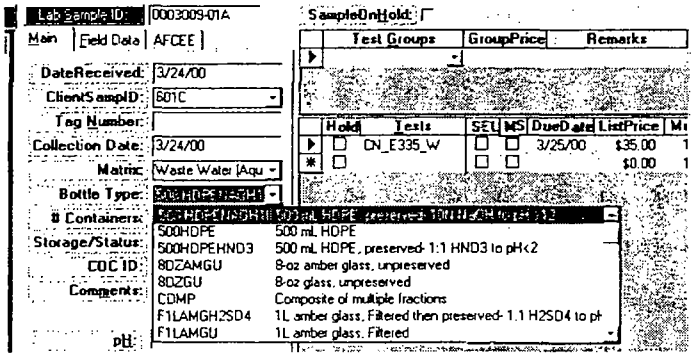


Fig. 3



Date received will carry over from other screen. Click on each of the following boxes to fill in the **Client Sample ID, Collection Date, Matrix, Bottle type**, and **Number of Containers** according to the COC (Fig. 4, Fig.3).

Fig 4



6.5.7 Read the login comments for specific guidance that may be required to log the correct test information for specific projects.

6.5.8 To choose the parameters either click on **Test groups or Tests** (Fig. 3) a list of possible parameters appears. Pick the appropriate test method for the appropriate matrix by clicking twice. If the sample is a wastewater choose W, if it is a solid choose S etc. If there is only one parameter and one bottle for the first sample ID click on **Add Sample** (Fig. 3) to get a new sample number. Then repeat above process.
If there is more than one parameter or bottle type for the first sample ID click on **Add Fraction** (Fig. 3). This feature will allow for the same number (since it is the same sample) but add a letter at the end to differentiate between the bottle types and parameters. If there are any test specific comments on the COC

these can be entered in the **Comments** field on the left or on the test line under **lab comments** column that is to the right of the test name. Then repeat above process as needed.

6.5.9 It is possible to copy tests if a different sample ID requires the same parameters. Click on add sample then fill in the Client ID and the collection date. Next click on **Copy Sample**, the computer will copy the information entered for the entire sample (including fractions and collection date). Similarly, if you want to copy a single fraction click on **Copy Fraction** (this only copies test information).

6.5.10 To put a sample on hold, log in samples for parameters listed and click **Sample on Hold** (Fig. 3). Sample will remain on hold until it is taken off by clicking on the same box.

6.5.11 To print labels click on **Labels**. The label screen will appear, Select the sample ID's that need to be printed by clicking the red arrow, when done click on **ok** and the labels will print.

6.5.12 When finished click the door on the upper right side until you reach the main work order screen. Repeat with a new client.

7.0 **Chemical Preservation and Storage:** Check and document chemical preservation of all preserved samples.

NOTE: Samples for volatile analyses (test codes: 624, 8260, 8021, 8015G) arriving in VOC vials should never be opened. The preservation of these samples is checked after analysis.

7.1 Check pH of wastewater samples for PCB/Pesticides analyses by method 608. If the pH of the sample is outside of **5.0 to 9.0** range, mark pH on the lid of the container and enter a note in the **Comment**.

7.2 Using a disposable transfer pipet, test pH by placing a drop of the sample on a strip of pH paper. *Do not dip the pH paper into the jar.* Preserve samples if the check shows that the samples were not preserved or if not enough preservatives was added.

7.3 If you have non-preserved sample for multiple tests requiring different chemical preservations, you may need to split sample and preserve each sub-sample separately.

7.4 Acids can be added using a disposable transfer pipet. Usually 2 ml of concentrated acid is enough to bring pH of sample to <2.

7.5 Addition of 2-3 pellets of Sodium hydroxide to 1L of sample is as a rule enough to bring pH to >12. Replace the cap and mix sample by inverting the jar. Test the sample again after addition of preservative.

Parameters	Preservatives	Color tape (to indicate used preservatives)
Ammonia	pH<2 H_2SO_4	Yellow
COD	pH<2 H_2SO_4	Yellow
Cyanide	pH>12 NaOH	Green
Hardness	pH<2 HNO_3	Red
TKN	pH<2 H_2SO_4	Yellow
Metals (except Chromium VI and dissolved metals)	pH<2 HNO_3	Red
FOG	pH<2 HCl	Blue
TOC	pH<2 H_2SO_4	Yellow
TRPH	pH<2 HCl	Blue
Phenols	pH<2 H_2SO_4	Yellow
Phosphorus	pH<2 H_2SO4	Yellow
Sulfide Total	pH>9 NaOH, ZnAc (if sample is	

	not analyzed immediately)	
PCBs/Pesticides	NaOH or H_2SO_4 if pH is out of range: 5<pH<9	

NOTE: If the sample was measured to have a pH > 10 and it requires tests that are preserved with acid then contact your immediate supervisor for further instructions. More importantly do not preserve the sample with acid because if it does contain CN then acidifying the sample will release CN gas.

7.5 Samples should not be preserved when the following known conditions exist:

- Sample has a pH <2 or >10
- Sample might produce toxic or explosive gases when preserved
- Sample is taken from an extremely hazardous waste site that is known to contain highly toxic materials.

7.6 Logged in samples are placed on appropriate shelves in the login cooler for storage at 2-6^{0}C. Once analysis has been started the samples may move to various coolers based on the analyses performed.

Samples for Wet Analysis	Cooler # 7
Samples for Org. Analysis	Cooler # 2
VOC samples (including BTEX)	Cooler # 4
Drinking Water for Metals	Cooler # 5
Drinking Water for Volatiles	Cooler # 10
Soil Samples for Volatiles	Cooler # 11
Ground Water for Volatiles	Cooler # 12; 13; 14

7.7 BOD samples are given directly to the BOD operator. **"Rush"** samples and samples that have parameters with very short holding times should be left in the login area and the appropriate supervisor must be notified.

Parameters	**Holding Time**
Hexavalent Chromium	24 hr
BOD	48 hr from the moment of the sample collection.
Residue Settleable	48 hr
Hydrogen Ion (pH)	Analyze immediately
Nitrate	48 hr
Nitrite	48 hr
Orthophosphate	48 hr
Surfactants	48 hr
Turbidity	48 hr
Chlorine Residual	Analyze immediately
Sulfite	Analyze immediately
Oxygen Dissolved	Analyze immediately

8.0 **Preparation of Composite Samples:** Volatile samples are not composited by the log-in technician. *Please see the Volatile Supervisor if such a request is made. Collect individual portions of the sample in a* clean stainless steel bucket or Pyrex dish and mix thoroughly using stainless steel spoon. Transfer obtained composite sample into jars and preserve if necessary.

8.1 When samples arrive they need to be in containers that can fit on shelves in the various coolers. Samples may come in buckets, boxes, bags and other forms. This will also include light bulbs, brake shoes, bricks and wood blocks. Break and mix sample as well as possible. The tools for breaking the samples into

appropriate sizes are provided. Put sub-samples into containers, label and place on the correct shelf. This is done to increase efficiency in the lab, allowing chemist to perform the analysis in the prescribed time frame.

8.2 Occasionally projects arise and clients request pre-preserved jars and coolers. Refer to Appendix #1 for appropriate container. The Login supervisor will determine what time constraints are involved and make arrangements for fulfilling such requests.

9.0 Reference:

9.1 SW-846. Chapter II; III; IV Revision 4, Nov. 2000.
9.2 2003 NELAC Standard. Quality Systems. 5.5.8 "Handling of Samples"

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Approved by: Mitchell Ostrowski 12/1/05

Appendix 1

40 CFR p. 136.3 Wastewater

Table II_Required Containers, Preservation Techniques, and Holding Times

Parameter No./name	Container	Preservation	Maximum holding time
Table IB Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	do	Do.
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PFTE, or Quartz	HNO_3 to pH<2	6 months.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	.do	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH >12, 0.6g ascorbic acid	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO_3 to pH<2,or H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen.	P, G	Cool, 4°C, H_2SO_4 to pH<2.	28 days.
Metals:			
18. Chromium VI	P, G	Cool, 4 °C	24 hours.
35. Mercury	P, G	HNO_3 to pH<2	28 days.
3, 5-8, 12,13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals except boron, chromium VI and mercury	P, G	HNO_3 to pH<2	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH<2.	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HC1 or H_2SO_4 or H_3PO_4, to pH<2.	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately.
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2.	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2.	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	do	7 days.
55. Residue, Nonfilterable (TSS).	P, G	do	7 days.
56. Residue, Settleable	P, G	do	48 hours.
57. Residue, volatile	P, G	do	7 days.
61. Silica	P, PFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	do	Do

Parameter No./name	Container	Preservation	Maximum holding time
65. Sulfate	P, G	do	Do
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P ,G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC Organic Tests			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons.	G, Teflon-lined, septum.	Cool, 4 °C, $Na_2S_2O_3$ 0.008%	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons.	G, Teflon-lined, septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$, HCl to pH2.	Do.
3, 4. Acrolein and acrylonitrile.	G, Teflon-lined, septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$, adjust pH to 4-5.	Do.
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols	G, Teflon-lined cap	Cool, 4 °C, 0.008% $Na_2S_2O_3$	7 days until extraction; 40 days after extraction
7, 38. Benzidines	G, Teflon-lined, septum.	do	7 days until extraction.
14, 17, 48, 50-52. Phthalate esters.	G, Teflon-lined, septum.	Cool, 4 °C	7 days until extraction; 40 days after extraction.
2-84. Nitrosamines	G, Teflon-lined, septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$, store in the dark.	Do.
88-94. PCBs	G, Teflon-lined, septum.	Cool, 4 °C	Do.
54, 55, 75, 79. Nitroaromatics and isophorone	G, Teflon-lined, septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$, store in the dark.	Do.
1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons	G, Teflon-lined, septum.	do	Do.
15, 16, 21, 31, 87. Haloethers	G, Teflon-lined, septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$	Do.
29, 35-37, 63-65, 73, 107, Chlorinated hydrocarbons	G, Teflon-lined, septum.	Cool, 4 °C	Do.
60-62, 66-72, 85, 86, 95-97, 102, 103. CDDs/CDFs.			
aqueous: field and lab preservation	G	Cool, 0-4 °C, pH<9, 0.008% $Na_2S_2O_3$	1 year.
Solids, mixed phase, and tissue: field preservation	G, Teflon-lined, septum.	Cool, <4 °C	7 days.
Solids, mixed phase, and tissue: lab preservation.	G, Teflon-lined, septum.	Freeze, <-10 °C	1 year.
Table ID Pesticides Tests:			
1-70. Pesticides	G, Teflon-lined, septum.	Cool, 4°C, pH 5-9	Do.

SW-846 Ground Water and Solid Waste

TABLE 4-1.
SAMPLE CONTAINERS, PRESERVATION, TECHNIQUES, AND HOLDING TIMES

VOLATILE ORGANICS			
Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	Method 5035: 40-mL vials with stirring bar. Method 5021: See method. Methods 5031 & 5032: 125-mL widemouth glass container. Use Teflon-lined lids for all procedures.	Cool to 4 °C.	14 days
Aqueous Samples With No Residual Chlorine Present	Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps	Cool to 4 °C and adjust pH to less than 2 with H_2SO_4, HCL or solid $NaHSO_4$.	14 days
Aqueous Samples WITH Residual Chlorine Present	Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps	Collect sample in a 125-mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial. Cool to 4 °C and adjust pH to less than 2 with H_2SO_4, HCL or solid $NaHSO_4$.	14 days
Acrolein and Acrylonitrile in Aqueous Sample	Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps	Adjust to pH 4-5. Cool to 4 °C.	14 days
Solid Samples (e.g. soils, sediments, sludges, ash)	Method 5035: 40-mL vials with septum and stirring bar. Method 5021: See method. Methods 5031 & 5032: 125-mL widemouth glass container with Teflon-lined lids.	See the individual methods.	14 days

SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES/PCBs AND HERBICIDES			
Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	125-mL widemouth glass with Teflon-lined lid	None	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.
Aqueous Samples With No Residual Chlorine Present	1-gal., 2 x 0.5-gal., or 4 x 1-L amber glass container with Teflon-lined lid	Cool to 4 °C	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.
Aqueous Samples WITH Residual Chlorine Present	1-gal., 2 x 0.5-gal., or 4 x 1-L amber glass container with Teflon-lined lid.	Add 3-mL 10% sodium thiosulfate solution per gallon or 0.008%. Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4 °C.	Samples extracted within 7 days and extracts analyzed within 40 days following extraction.
Solid Samples (e.g. soils, sediments, sludges, ash)	250-mL widemouth glass container with Teflon-lined lid	Cool to 4 °C	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.

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SAMPLE HOLDING TIMES, RECOMMENDED DIGESTION VOLUMES AND RECOMMENDED COLLECTION VOLUMES FOR INORGANIC DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digestion Volume, (mL)[1]	Collection Volume (mL)[1]	Treatment[2] Preservative Holding Time[3]
Inorganic Analytes (except hexavalent chromium and mercury):			
Aqueous			
Total	100	600	HNO_3 to pH <2 6 months
Dissolved	100	600	Filter on site: HNO_3 to pH <2 6 months
Suspended	100	600	Filter on site 6 months
Solid			
Total	2 g	200 g	6 months
Hexavalent Chromium:			
Aqueous	100	400	24 hours Store at 4 ± 2 C until analyzed
Solid	2.5 g	100 g	One month to extraction, 4 days after extraction Store at 4 ± 2 C until analyzed
Mercury:			
Aqueous			
Total	100	400	HNO_3 to pH <2 28 days
Dissolved	100	400	Filter: HNO_3 to pH <2 28 days
Solid			
Total	0.2 g	200 g	28 days Store at 4 ± 2 C until analyzed

[1] Unless stated otherwise.
Either glass or plastic containers may be used.
Any sample volume reduction from the reference method's instructions must be made in the exact proportion as described in the method and representative sampling must be maintained.

TITLE: VALIDATION OF DATA

KEY WORDS: Not applicable.

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY:	Brian Goyette	March 7, 2008

APPROVALS:

QUALITY ASSURANCE MANAGER:	Brian Goyette	March 7, 2008
QUALITY ASSURANCE MANAGER:	Mary Lubitov	March 7, 2008

VALIDATION OF DATA

1.0 SCOPE AND APPLICATION

1.1 The purpose of data validation is to verify the correctness of analytical results. As an outcome of validation process the data can be accepted and released to the client or it can be rejected.

1.2 The data validation is conducted by a qualified designated personnel: a second analyst, not involved in the analysis of the sample which data is in question, Lab. Director, Department Supervisor, or the QA *department.*

1.3 The data reviewer must report to the Lab. Director or *QA staff* any problems and non-conformities concerning the data.

2.0 TERMINOLOGY

Refer to EMT "Quality Manual" for terms used in this SOP.

3.0 PROCEDURE

3.1 The set of criteria to be checked *initially by the analyst*:

3.1.1 Calibration, initial and continuing.

3.1.2 Data Handling; *verify calculations, analyte identification, integrations*

3.1.3 Duplicate *analysis*

3.1.4 Holding times

3.1.5 Interference Check Standard (Metals -ICPs)

3.1.6 Internal Standards

3.1.7 Laboratory Control Samples (LCS)

3.1.8 Matrix Spike *(MS), Matrix Spike Duplicate (MSD).*

3.1.9 Method Blanks

3.1.10 Surrogates – *for organic analysis*

3.2 Calibration

3.2.1 Instrument capability to produce credible data should be demonstrated by an acceptable initial calibration and subsequent calibration verification.

3.2.2 To be accepted the calibration should include a minimum number of points, required by the specific method, the standards should be evenly distributed.

3.2.3 The coefficient of correlation is to be > 0.995 for inorganic analyses, and > 0.99 for organic analyses.

3.2.4 Zero calibration setting should be in position "ignored" for organic analysis when a least

squares regression is used.

3.2.5 Refer to the specific SOP for more information on a particular method or instrument calibration requirements.

3.3 Initial and Continuous Verification.

3.3.1 *Organic methods require the analysis of a Continuing Calibration Verification Standard (CCV) to be run at the beginning of every analytical sequence and every 12 hours minimum. The recovery of all compounds of interest should be validated against the limits specified by the appropriate method or those developed in house. Default CCV recovery limits used in the laboratory are 80-120% for GC/MS analyses, 85-115% for GC and HPLC analysis. Surrogate compounds in the CCV are evaluated using the same criteria as for the analytes. For multi-analyte organic methods such as method 8260 VOA's it is not unusual to have a few analytes outside of the control limits, these outliers must be noted in the laboratory narrative. Up to 20% of target analytes may be outside control limits. However, if a target analyte in a CCV has a recovery below limits and that analyte is present in an associated sample the sample must be re-run with an acceptable CCV. If a CCV has compounds outside of control limits but those compounds are not present in the associated samples the data may be reported. If several target analytes are below recovery limits, recalibration may be necessary or the CCV may need to be re-prepared. Consistent recoveries either below or above control limits may indicate the need to recalibrate or a degraded CCV standard.*

3.3.2 Inorganic Lab. Check standards should be analyzed in the beginning, at the end of the analytical run and every 10 samples. Recoveries of 90-110%, or *those specified by the method* should be obtained as a minimum to consider the calibration verified. Low Level Check standards employed for some tests (Mercury by VGA, Lead in DW and etc.) are evaluated using the limits developed by the laboratory. If the Check Standard recovery does not comply with the limits the data is unusable.

3.4 Data Handling. The raw data should be examined *by the analyst* for:

3.4.1 Complete Sample Information: sample number, date of preparation and date of analysis.

3.4.2 Any anomalies: carryover, baseline shifts; negatives, abnormal peaks' shape, peak tailing and etc.

3.4.3 Correct Compound Identification. *During secondary review, by either a senior analyst or supervisor,* approximately 10% of samples should have all compound identification verified, verify sample calculations. The confirmation technique should be used if the compound identification is in a question. *Second column confirmation is* required always for DW by HPLC and for Pesticides by GC.

3.4.4 Transcription or reduction errors (mistakes in calculations involving dilution, concentration, correction for sample weight, % solids); correctness of rounding; appropriate number of significant figures. *For these kinds of errors the department supervisors or the QA group kinds should check 10% of reported results.*

3.4.5 Analytes with concentrations falling outside of calibration range.

3.4.6 Reporting limits reflecting dilutions, interferences, and amount of sample used.

3.4.7 Traceability for standards and spikes.

3.4.8 Adequate explanation of corrective actions if any.

3.4.9 Correct handling of wrongly entered data.

3.4.10 Identifiable signatures or initials of analysts involved in analysis.

3.5 Duplicate.
Duplicate analyses are indicators of laboratory precision based on sample matrix. Inorganic Analysis: For the sample with concentration > 5 x MDL the RPD limit is 20% or less, if based on the laboratory performance data. Organic Analysis: The RPD limits based on the laboratory performance data are to be used for the data validation. RPDs for organic analysis are *typically generated from the analysis of MS/MSD samples.*

3.6 Holding time.
Actual holding time is established by comparing the sampling date with the date of analysis or the date of the sample preparation. Date of the sample collection can be found on the worksheet *generated by the LIMS*, the date of preparation is in the appropriate digestion/extraction books; the date of analysis is on the raw data. Sample preservation to the proper pH should also be checked. Information about the sample preservation can be found on COC.

3.7 Interference Check Standard.
Interference Check Standard is analyzed by the ICP to verify interelement and background correction factors. IEC factors used for each analytical run should be available. IEC standard has to be analyzed at the beginning of each day of analyses. The result for the IEC solution must fall within the range: +/- 2.2 x MDL for analyte of interest and with +/- 20% for the interfering element.

3.8 Internal Standard.
Internal Standards used in organic analysis are used to monitor *instrument conditions such as correct and consistent injection volumes and instrument sensitivity.* Changes in internal standard response will adversely affect quantification of target compounds. Internal Standard area should be within method specified limits for all quality control samples and regular samples, including sample dilutions and reanalysis. If the internal standard response is outside the upper or lower limits the data is unusable and the sample should be re-analyzed. *If reanalysis gives the same results re-extract the samples.*

3.9 Laboratory Control Sample (LCS).

9.2.1 LCS monitors overall performance of all steps in the analysis, including the sample preparation. All LCS results must fall within the control limits established by the laboratory or specified by the method whichever is more stringent. If LCS recovery falls within the method specified limits but outside of the laboratory limits the problem should be investigated and evaluated. It is the prerogative of the Lab. Director to decide whether the data can be reported or not. If LCS recovery does not comply with the method specified limits the data is unusable.

9.2.2 *A LCS is most common in organic analysis; it typically contains all analytes of a particular method.* The LCS monitors the overall performance of all steps in the analysis, including *sample extraction and concentration.* LCS recovery limits are typically generated in the laboratory by taking the average recovery of a set data. *For multi-analyte organic methods such as method 8260 VOA's it is not unusual to have a few analytes outside of the control limits, these outliers must be noted in the laboratory narrative. Up to 20% of target analytes may be outside control limits. If more than 20% of analytes exceed limits re-extraction may be required.*

3.10 Matrix Spike (MS), Matrix Spike Duplicate (MSD).

3.10.1 MS and MSD analysis provide information about the *effect of sample matrix on sample extraction, concentration, and the identification and quantitation of target analytes, and also the homogeneity of the sample.* Matrix spike recoveries and the calculation of relative percent differences between the duplicate samples are evaluated against the limits established by the laboratory or those specified by the method.

3.10.2 *For multi-analyte organic methods such as method 8260 VOA's it is not unusual to have a few analytes outside of the control limits, these outliers must be noted in the laboratory narrative. Up to 20% of target analytes may be outside control limits. A matrix spike with poor recoveries should be re-analyzed to verify matrix as the cause of the poor recoveries.* If recovery of any analyte falls outside the control limits and the LCS for the analyte is shown to be in control, the recovery problem encountered with the spiked sample is judged to be matrix related not system related.

3.10.3 *If the RPDs for several analytes are outside of the required limits sample matrix or non-homogeneous sample matrix is a possible cause. If more than 20% of analytes exceed limits re-extraction may be required if sample matrix cannot be proven as the cause of the outliers.* Metals Analysis.

3.10.4 Serial Dilution Test should be done when physical or chemical interferences due to the matrix is suspected. The test is conducted for the samples with sufficiently high analyte concentration (20 x MDL or above). The matrix specific interference is considered to be confirmed if the result of 5-fold diluted sample differs more than 10% of the original result.

3.10.5 Post digest spike is used for the samples with matrix specific problems and low analyte concentration. The spike concentration should be 10-20 times MDL and more than 50% of the background concentration, whichever is higher; the volume of spike should be no more than 10% of the sample's analytical volume.

3.10.6 MSA is conducted for samples with matrix specific problems and post preparation spike recovery above 40%. The standards used for MSA should be 50%, 100% and 150% of the sample concentration. MSA slope should differ by no more than 20% of the calibration curve slope. Coefficient of correlation for MSA should be > 0.995. If the requirements listed above are not met the data is qualified as estimated.

3.11 Method Blank.

3.11.1 Blank evaluation is required to determine the existence and magnitude of contamination problem. Absolute Value of the Method Blank should be less than MDL, or 5% of the regulatory limit for the analyte for solid waste and 10% for water, or 5% of the measured concentration in the sample for solid waste and 10% for water.

3.11.2 Method Blank should be run with batch of 20 or less samples. In instances where more than one blank is associated with a sample, the qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant.

3.11.3 *The analysis of a calibration blank is required both in the beginning, and at the end of the run, and after every 10 samples for inorganic tests. Solvent or method blanks should be analyzed with each analytical set of samples for organic analyses. Any target analytes found in the blank should be less than the PQL. The presence of target analytes in the blank may indicate contamination. If target analytes are present in the blank inspect the analytical system to identify the source of the contaminants so it may be eliminated. The contamination may be from carry over from a previously run sample, contaminated standards, or contaminated glassware etc. Clean all glassware, re-prep standards, and re-prep samples and associated QC. Method Blank contamination in organic methods may require the re-extraction of samples and all associated QC. In some cases if target analytes are present in the blank but all associated samples are free of target analytes raise the reporting limit in the sample to just above the contamination level and report the data. In organic methods if contamination is serious, notify the organic prep group leader and the QC group to initiate an investigation and the corrective action process.*

3.12 Surrogate.

3.12.1 Surrogate recovery in the individual samples is evaluated versus the limits based on historical laboratory data or the method specific limits. 70-130% limit can be used as interim acceptance criteria if not enough data is accumulated to develop the laboratory limits and the methods do not contain method specified recovery limits.

3.12.2 The samples with surrogate recovery outside of acceptable limits should initially be re-analyzed *to verify the recovery. If re-analysis produces the same results the sample should be re-extracted and reanalyzed. If re-extraction and reanalysis produces the same results the outliers are assumed to be a result of the sample matrix a comment is added to the laboratory narrative indicating that surrogate outliers are due to sample matrix.* In some samples requiring dilution the surrogate cannot be measured. In these cases the surrogate recovery from the undiluted sample, if available, should be provided.

3.13 *Any violation of the method requirements or procedures outlined in this document may render the data unreportable. Repreping or re-extracting samples may correct outliers or verify matrix as the cause of QC violations. All violations or QC outliers should be well documented in the laboratory narrative.*

3.14 After successful completion of the validation steps the data is to be singed and dated and approved for submittal.

4.0 REFERENCES

4.1 EPA. Laboratory Data Validation Functional Guidelines for Evaluation Inorganic Analyses.

4.2 *Laboratory Quality Assurance Manual, Environmental Monitoring and Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.*

4.3 SW-846, 3D Edition. Revision 1. Chapter One.

4.4 SW-846, 3D Edition. Revision 1. Method # 8000.

TITLE: REPORTING AND MANAGING RESULTS IN *LIMS*

KEY WORDS: Not applicable.

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY: Matt Gregory November 5, 2008

APPROVALS:

TECHNICAL MANAGER: Matt Gregory November 5, 2008

QUALITY ASSURANCE MANAGER: Brian Goyette November 5, 2008

REPORTING AND MANAGING RESULTS IN *LIMS*

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures and practices that must be followed in order to report complete and accurate information to the *LIMS*.

1.2 This is a guideline for all analysts who enter data into *the* Omega *LIMS system*.

1.3 *Not every example of data uploading is covered in this SOP, as many instruments also require secondary software to link the instrument to the LIMS. Refer to the specific method SOP's for the very specific information required for some methods and instrumentation.*

2.0 TERMINOLOGY

2.1 *Refer to EMT "Quality Assurance Manual" for terms used in this SOP.*

3.0 PROCEDURE

To create a new data table for preparation or analytical information entry one will select "Prep" for a prep batch or "Data Entry by Run" for the data entry (see figure one for the main screen of the Omega LIMS starting point).

3.1.1 *To create the prep batch: select "Prep" from main screen, click "Add" from top toolbar (automatically creates the new table and unique prep sequence ID number), select the test code needed from the prep code drop down list, modify start date and technician as needed, select "LoadSamps" followed by "User" to get sample selection, and select samples by highlighting the work order numbers and splits in left column and using right arrow to move to right column for the selected samples. Click the "OK" when all needed samples are in the right column; samples will be automatically put into the table with a Method blank and an LCS (additional QC types must be manually typed in, see 3.2.2 or MBLK and LCS line deleted if not needed or applicable). Enter all needed initial weights or volumes and modify final volumes if different than the default. To close and save the prep table enter the final date and time (End date) that will also lock the information. After the final date is entered, click the "back page icon" to exit followed by clicking "Yes" to the dialog box query that will appear asking "Update sample PREP status to Complete?".*

NOTE: *The prep batch must always be created and saved (closed by putting in the batch completed date) before the analytical data is entered for the system calculations, validations, and QC linkages to work properly.*

3.1.2 *To create and start the data table,* open "Data Entry By Run" *and select "Add" from toolbar to add a new record that will create a new data sequence.* Enter the *"Instrument ID" (manual, ICP-MS, GC-MS, etc), Run start date (when the analysis was started, not necessarily ended) and* analyst's name; making sure that the information in Omega is consistent with the information on the instrument report *or in the lab notebook.* Enter the time of analysis for tests with a holding time less than or equal to 48 hours such as: pH, Hexavalent Chromium, Sulfites, Sulfides, BOD, Residual Chlorine, etc. *See figure 2 for location of items on screen.*

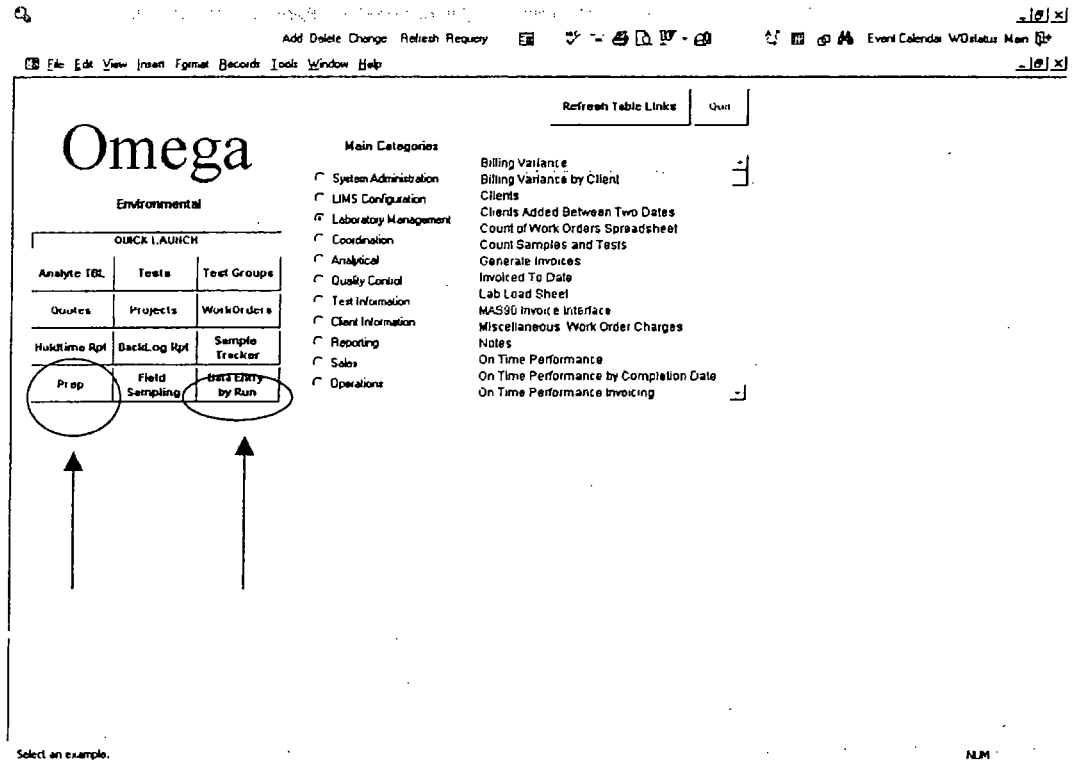


Figure 1: the Main screen of Omega displaying the starting points for the creation of a prep table or analytical data table (section 3.1).

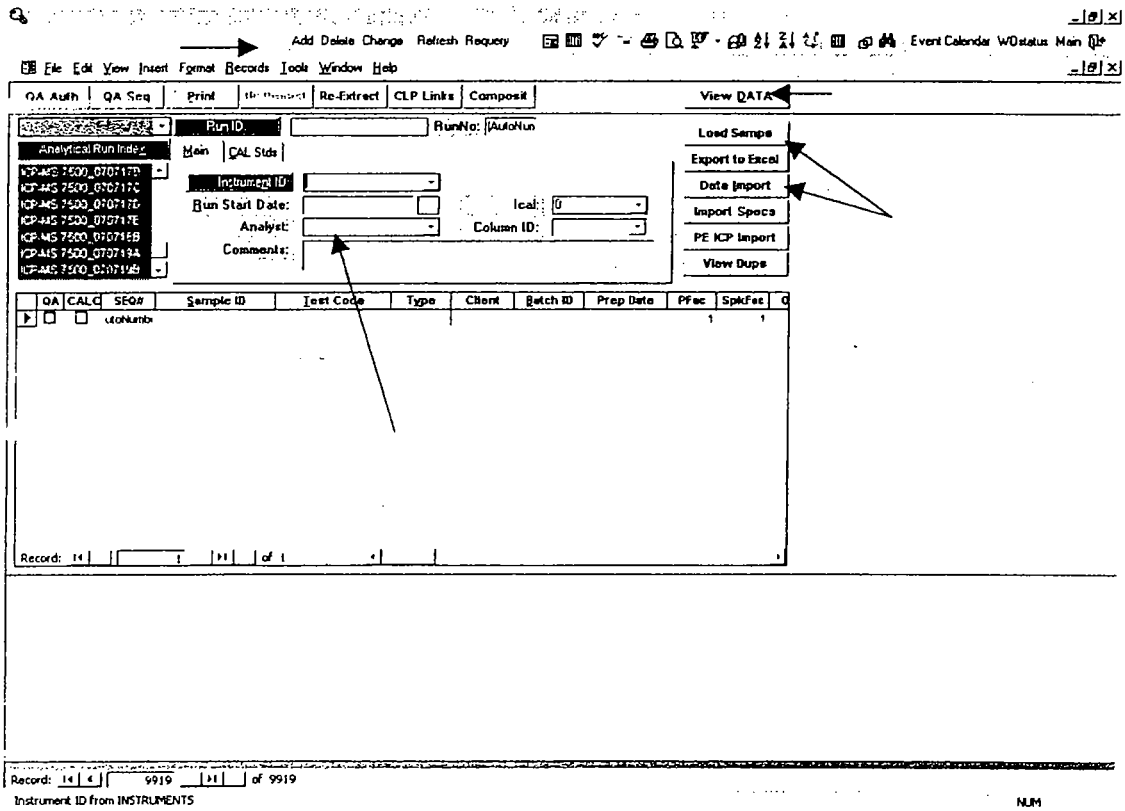


Figure 2: the first page of the data entry table showing the various data entry boxes and tabs needed for various applications (see sections 3.2 to 3.8 for proper use).

3.2 *Bring* the samples *into the table* either by importing the data from instruments, by loading the samples from Omega *by test code or prep sequence*, or by *manually* typing in the samples' *work order* numbers. *These operations are performed using the "Load Samples" (to manually select from all samples logged in for a certain test code or from a prep sequence) or "Data Import" to upload instrument data. Refer to the specific tests for instrument import specifications. Generally if all is set correct at the instrument file level all you need to do after clicking the "Data Import" tab is to find the file and open, all tables will be created and data will load automatically. To select form test codes or prep sequences select the test code needed or type in the prep sequence in the available line after clicking the "Load samples" tab. Move the needed samples from the left column to the right by highlighting and using the right arrow tab. After all are selected, click "OK" to create the data table. Make sure that when pulling in QC from a prep sequence that the test code and sample type are present (may need to type in the needed information) or the data load will fail for the QC samples and they will not be created in the table. They can be manually typed in with all needed information selected across the horizontal line as an alternative method of creating the sample in the table.* Only samples analyzed by the same test code should be on each data entry page *to make QC reports link to the QC samples correctly.*

3.2.1 Water and soil samples processed together in the same analytical run and shar*ing* the same check standard *can be loaded into the same table with some restrictions.* They must be entered in separate sets *(batches)*, each with its own QC and corresponding test code. *The QC is linked by the prep sequence (where applicable) and /or test code.*

3.2.2 Identify QC sample *types* as follows (typical sample examples):

Matrix spike:	01040515-01AMS
Matrix spike duplicate	01040515-01AMSD
Matrix duplicate:	01040515-01ADUP
Post-digestion spike	01040515-01APDS
Post-digestion spike duplicate	01040515-01APDSD
TPH, *BOD, COD* QC *only* sample:	01040515-01AQC
Lab control sample *(LCS)*:	LCS-4625 or LCS-R19950
Method blank (MB*LK*):	MB-4625 or MB-R19950
TCLP *or ZHE* blank:	MBT-4624 or MBZHE-4625

(The numbers are used only as examples)

3.2.3 *An example of a* "QC sample" identifies 8015 TPH samples (in the above example) that contain something other than diesel and were integrated with the Diesel method. This sample is used for matrix spike recovery calculation only. *The "QC" can also be used for BOD, COD, and others where a value needs to be subtracted for the MS/MSD calculation, but the result is not to be reported out as the final result to the client on the report. Most likely the "QC" only sample has a proportional value to the final result due to a dilution from adding the volume of the spike [COD] or is not a total average [BOD].*

3.2.4 Including the batch number in the identification of the MB and LCS links the QC to its prep or analytical batch. The MB and LCS should be named as above on the instrument reports transferred to Omega.

3.2.5 TCLP Blanks: keep the number of the TCLP batch when they are analyzed for organic parameters. When analyzed for inorganic parameters, TCLP (or SESW) blanks receive the number of the analytical or preparation batch with which they were processed.

3.3 "Test Code" indicates the analytical method and the matrix type. For example, 8270_W is *the* method 8270 for waters *and* 8270_S is 8270 for soils. Use the same Test Code for samples as well as associated control samples (CCV, Blanks, LCS, MS/MSD). *Test codes are named starting with the Method number (from EPA, ASTM, Standard Methods, etc.) followed by a very brief description (for some) and then the matrix at the end.*

NOTE: *Updated test code lists for the Omega LIMS can be found on the network at: "G:\Subcontract info & Omega test lists".*

3.4 *Properly identify* the type of sample:

SAMP	Regular sample type to be reported to client
MBLK	Method blank
ICV	Initial calibration verification
ICB	Initial calibration blank
CCV	Continuous calibration verification
CCB	Continuous calibration blank
LCS	Laboratory control sample
MS	Matrix spike
MSD	Matrix spike duplicate
PDS	Post digestion spike
PDSD	Post digestion spike duplicate
DUP	Duplicate

3.4.1 CCV1, CCV2... and LCS1, LCS2...are for tests in which different levels of check standard or spiking solution are analyzed. In the organic lab, for TCLP and ZHE blanks choose SAMP as the sample type.

3.5 "Batch ID" refers to either the preparation or analytical batch (sequence) number. Change the "Batch ID" entry for the CCV (or other instrument QC not generated by the prep) to match the batch IDs of the samples. Since the " batch ID" cell cannot admit more than two batch numbers, the CCV cannot be associated with more than two batches. *The same CCV results may need to be entered into a data table for mixed runs where more than one test code or prep are involved so that all samples will be able to link to the associated QC if a QC report is needed and generated.*

3.6 "Pmoist" is needed for results based on *the* dry solid content *that will show as the tests units-dry when Pmoist correction is applied.* The number represents the percentage of moisture. This value must be entered into Omega before the samples are calculated. *If the Pmoist results are already entered the values will automatically be pulled into the data table when the samples are brought in.* For rush samples if the percent moisture is not ready *(reported)*, report the results on a wet *weight* basis and recalculate later, when the percent moisture *result* is available.

3.7 Enter dilution factors in the "DF" column when samples are diluted *at the bench* after preparation or when a reduced sample size is used for analysis *(VOC)*. Only use dilution factors when it is in addition to the initial and final amounts used in the prep sequence. *For example, a sample over-ranges the calibration by a factor of five during analysis. The sample was diluted by taking 1 ml of original to 10 ml of DI (1 to 10), so a "10" needs to be put into the "DF" column.*

3.8 The sequence numbers in BLKref, SPKref and RPDref columns link quality control with regular samples. When entering data, always make sure samples refer to the correct QC. In other words, make sure that regular samples have the proper BLKref, CCVref, duplicates, and matrix spike duplicates refer to the correct RPDref; and matrix spikes have the right SPKref. The majority of mistakes occur in the references to instrument QC. Make sure that all QC refers to the correct analytical run sequence. For example, for purge and trap analysis the BLKref should be the most recent blank analyzed before the sample. Omega automatically provides the references within the preparation batch, but only when the reported data contains no identification errors and when the samples are loaded in the certain sequence. Method blank should be loaded before LCS and the other samples of the batch, samples chosen for QC should be should be loaded before their matrix spike/matrix spike duplicate. Deviations from the required data entry sequence or errors in sample identification will result in missing or incorrect references. In some cases the reference will appear after the sample ID was corrected. Sometimes you will need to re-load the samples or re-calculate the sequence to re-link all of the information.

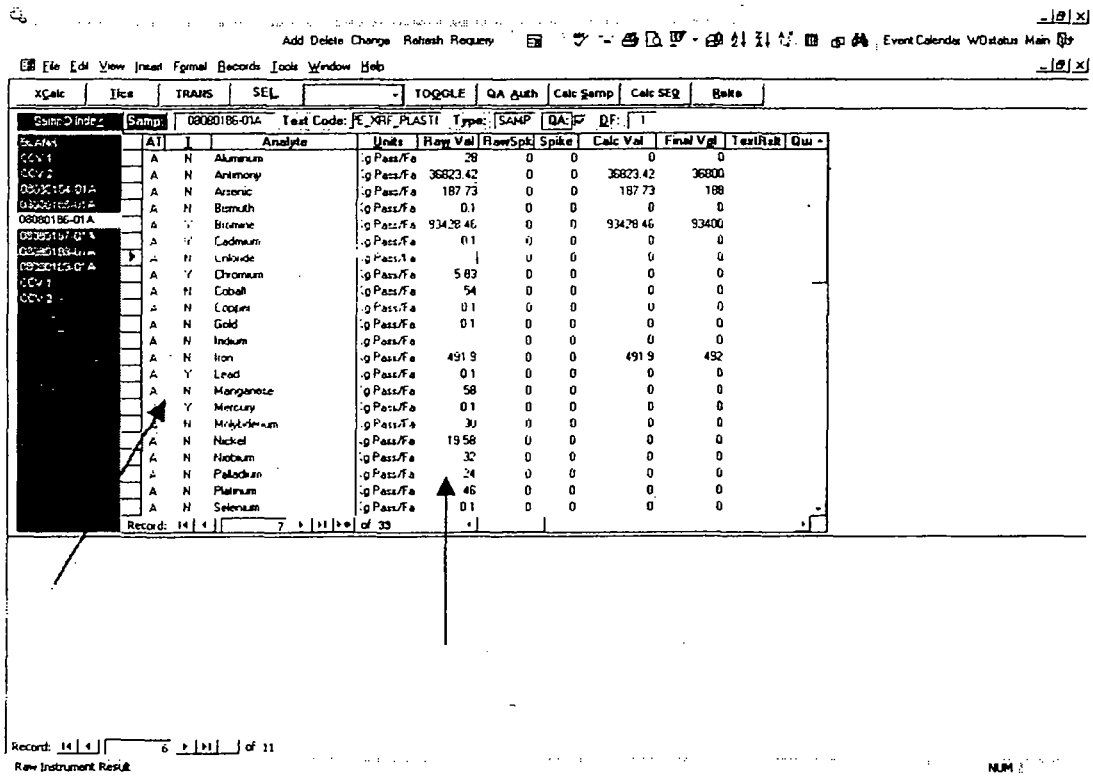


Figure 3: Omega data table "second page" after clicking "View Data" from first screen displaying the highlighted sample in use, the "T" column, and the raw result column (see sections 3.9 to 3.11).

3.9 *To manually report the raw data values select "View Data" from the top right to get to the tables to enter the actual numbers obtained. From instrument loading this will automatically be done. Select the samples created on the left side of the screen to be able to type in the coordinating results for each sample by double clicking the work order for result entry. When all needed data is entered, select "Calc SEQ" to have the system calculate the results to the final value, incorporating all prep information, dilution factors, and Pmoist corrections (if needed).* It is helpful to compare the results in Omega with the results in the bench book to verify the calculation. Check entered initial and final weights, volumes, dilution factors and raw values. If the data is transferred from an instrument, check the calculation of *at least* one analyte *in a multi-analytical sample entry.*

3.10 When samples are entered into Omega more than once due to multiple dilutions or other reasons, carefully check which analytes are marked "Y" and "N" in each run *in the "T" column.* Failure to mark compounds correctly can result in double reporting or underreporting, making the final report *incorrect or partially complete.* To facilitate the reporting of a sample from multiple runs, use the following procedure:

3.10.1 Load and calculate the sample from one run. Change "Y" to "N" for compounds that will not be reported from this run *in the "T" column. Data imported from an instrument using the "Data Import" tab will automatically assign the Y and N settings for the samples based on log-in. The Y and N values will need to be matched on the associated QC samples (LCS, CCV, CCB, etc.) or set to N for any analytes that need a re-run due a QC problem or needed dilution.*

3.10.2 *To find incomplete data due to re-runs* launch into "Work Order", click on the sample number and go to sample status. Double-click in the "COMP" *column* cell corresponding to the sample, and check the list of missing analytes. Toggling from this page to the data entry run *(by double clicking in the cell for the correct run sequence)*, check and report the rest of *the* analytes from the other runs, changing "Y" to "N" wherever necessary. Check your Backlog

Report for any samples with a mark in the PC (partially completed) column, which indicates that there are compounds missing from the analysis.

3.11 The "Qual" column *contains* several codes *that may require attention due to QC failures.* A "J" indicates that values are detected below PQL level *and above the MDL level*, an "H" appears if the samples are analyzed passed *the* Hold time, a "B" indicates method blank contamination above *the* PQL level, and an "S" or "R" mean that the spike recovery *(S), spike duplicate recovery (S)*, or RPD *(R)* are outside of the acceptable range. Appearance of the S or R qualifiers can be due to a data entry error and may disappear after correction of the mistake. If it does not, investigate the reason for the S or R and provide an explanation in the comments column *(if the data is reportable as some flags will make the data fail requiring a re-run, see 3.12 to 3.13.9)*, or see your supervisor.

3.12 The data reported in Omega must meet QC criteria: Evaluation of QC data includes evaluation of calibration, instrument performance, calibration verification, blanks, LCS, duplicates, matrix spikes and surrogates depending on which parameters in the sample have to be reported and which method is used. If QC meets acceptance criteria, all is well. If some compounds fail to meet QC criteria, examine how the failure applies to each particular sample. First, check whether the failed compounds are analytes of interest. No action or comments are needed if the failed parameters are not on the sample selection list. In some cases, data associated with failed QC results can still be reliable. Such data would be reported with full disclosure of QC results and their interpretation in the comment column. For example:

3.12.1 The data *is* valid if the level of contamination in the MB is less than 1/10 the level of the analyte in the sample (i.e., the contribution from the blank is less than 10%), or if the analyte is not detected in the sample.

The data *is* valid if recoveries of a target analyte are higher than normal in the check standard, but that analyte is below detection limit in the sample and reported as a non-detect.

3.13 If the Check standards and LCS analyzed with the batch meet QC requirements but the MS/MSD fails due to matrix interference, the problem relates only to the particular sample. An explanation in the comments column is necessary only for the sample used to set up MS/MSD. The "Notes" are available in Omega but they do not eliminate the requirement for clear and adequate interpretation of the results. The following are examples of acceptable comments:

3.13.1 Initial calibration meets 8081 acceptance criteria. 4,4-DDT and Endrin have RSD values exceeding 20%; however the average of the RSD values for all analytes is less than 20%.

3.13.2 The recoveries of 1,2-Dichlorobenzene and 1,3-Dichlorobenzene in the check standard are above the laboratory limit of 120%. Since the analytes are not detected in the samples the data are still considered to be valid.

3.13.3 The analytes found in the method blank are not detected in the sample.

3.13.4 The level of contamination in the method blank does not exceed the NELAC requirement of less than 1/10 of the measured concentration of sample (or regulatory limit).

3.13.5 The low/high surrogate recovery is due to a matrix-related problem. The reported results associated with the surrogate target compounds are considered to be estimated values.

3.13.6 The low/high matrix spike recovery is due to matrix interference. The LCS and check standards analyzed with the sample indicate no processing problems.

3.13.7 The method blank associated with samples 01040099-01A through 0140099-20A, which were extracted in one batch, was contaminated with the following PNA compounds: Acenaphthene, Anthracene, Fluoranthene, Fluorene, Pyrene and Phenanthrene. These analytes were not detected in the samples.

3.13.8 The average recovery for all analytes in the CCV is within 85-115%. The high recoveries of DDT (117%) and Endrin Aldehyde (143%) do not compromise the data since these analytes are not detected in the samples.

3.13.9 The recovery of DDT in the MS and MSD performed on sample 0103332-01F is low due to matrix interference (subtraction of the co-extracted materials from the spike value).

3.14 *Document encountered problems, corrective measures and the efficiently of corrective action as required per SOP # 247 "Corrective Actions".*

3.15 *When an original sample and duplicate are run in a batch, the original result should be the reported value on the final report. If a legitimate reason to question the original result arises due to an instrument issue, dilution, or other possible error the duplicate may be reported as long as the data is documented that the duplicate was reported with a reason for switching the result. Generally if there is a large difference between the original and duplicate the RPD will fail and the proper steps for a high RPD should be followed (see the method specific SOP quality control section).*

NOTE: *Always refer to the specific test SOP for specific QC requirements. The above examples are mainly for guidance purposes and do not fully cover all tests, accreditations, or client requirements.*

4.0 REFERENCE

4.1 *Laboratory Quality Assurance Manual, Environmental Monitoring and Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.*

TITLE: DOCUMENT CONTROL

KEY WORDS: Not applicable.

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY:	Brian Goyette	May 9, 2008

APPROVALS:

QUALITY ASSURANCE MANAGER:	Brian Goyette	May 9, 2008
QUALITY ASSURANCE MANAGER:	Mary Lubitov	May 9, 2008

DOCUMENT CONTROL

1.0 SCOPE AND APPLICATION

1.1 *Document Control procedures apply to the following documents: EMT Quality Assurance Manual, SOPs which include analytical, general operations, and field, training documents, corrective and preventive action reports, internal and external audit reports, laboratory bench log books, and other documents that are part of the EMT management system.*

1.2 *These procedures insure that all documents issued to personnel in the laboratory as part of the management system are reviewed and approved for use by authorized personnel prior to issuance.*

1.3 *A master document list is maintained which identifies effective dates, current revision status and distribution of documents in the management system. This document is readily available to preclude the use of invalid and/or obsolete documents.*

1.4 *Invalid or obsolete documents are promptly removed from all points of use to insure against unintended use.*

1.5 *Procedures exist to insure that authorized revisions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed.*

1.6 *Documents are periodically reviewed and, where necessary, are revised to ensure continuing suitability and compliance with applicable requirements.*

1.7 *All laboratory notebooks issued are numbered. The numbers are tracked so the current book or logbook in use can be determined. When the book is full or no longer in use the number is added to the archived data list and is placed in the current data storage box. All contents of the data storage box are identified so that the box the logbook is in can be located in the storage area if needed.*

1.8 *Revised or obsolete documents are archived, stored either electronically or as hard copies.*

2.0 TERMINOLOGY

Refer to EMT "Quality Assurance Manual" for terms used in this SOP.

3.0 PROCEDURE

3.1 Document control applies to several different types of documents at EMT, some include; SOPs, QAM, corrective action reports, instrument/equipment manuals, instrument run logs, and other documents. All documents in the laboratory are monitored and documented as described below.

3.2 *The revision, approval and distribution of SOPs in the laboratory follow strict procedures.*

3.2.1 *Each SOP has a unique number. Approximately every two years or, more frequent, if required to resolve issues raised during internal or external assessments or method updates, SOPs are revised.*

3.2.2 *Hand written changes to SOPs are not acceptable.*

3.2.3 *All changes or alterations to SOPs are italicized. These changes remain italicized until the next revision of the document.*

3.2.4 *Revised SOPs will have a new revision number and effective date. Only approved copies of SOPs are distributed in the laboratory.*

3.2.5 *Approved copies of SOPs are present in each laboratory area in a red binder. The original signed, controlled, copy of each SOP is kept on file in the QA office.*

3.2.6 *A Master Document list is maintained which tracks SOP history and lists the current revision and effective date for all EMT SOPs.*

3.3 *The Quality Assurance Manual is updated annually. All additions are italicized; these changes remain italicized until the next revision of the document. Each revision is issued with a new revision and effective date.*

3.4 *Corrective Action Reports.*

3.4.1 *Electronic copies are kept on file in the QA directory on the EMT intranet. Electronic copies are kept in the CAR or Corrective Action Report directory. This directory is then broken down by years.*

3.4.2 *A new procedure implemented in 2008 follows the following procedure:*

3.4.2.1 *All Corrective Actions will be identified by year, then numbered sequentially during the calendar year, the first report each year being number 1.*

3.4.2.2 *After numbering the source of the corrective action will be identified using the following convention: EA-External Audit, IA-Internal Audit, PT- Proficiency Test, QS-Quality System, MSC-Miscellaneous.*

3.4.2.3 *Example of the Registration Number Abbreviations: 08-001-EA - Year - orderly Number for the Year - Category of Corrective Action*

3.4.2.4 *There are two file folders under the 2008 file, they are "Opened 2008" and "Closed 2008"*

3.5 Instrument manuals are labeled with unique letters specific to each department then numbered incrementally. These manuals are located in specific defined areas within the laboratory or QA office. Please refer to the attached appendices for a list of manuals for each department.

3.6 *Instrument run logs or bench logbooks are also part of the document control system. All lab books issued to analysts are assigned a specific number. The numbers are tracked so that it is possible at all times to know which books are in use and which books have been archived.*

3.7 Other documents include: ISO 17025 documents, NELAC documents etc. They are labeled numerically and kept in the QA office.

3.8 *Archived documents are maintained for a minimum of 5 years.*

4.0 REFERENCES

4.1 Laboratory Quality Assurance Manual, Environmental Monitoring and Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.

4.2 NELAC, National Environmental Laboratory Accreditation Conference. Chapter 5, 2003 NELAC Standard 6/5/2003. Effective date 7/1/2005.

4.3 ISO/IEC 17025,General requirements for the competence of testing and calibration laboratories. ISO/IEC 17025:1999(E)

APPENDIX A
Metals Instrument Manuals

#	Instrument Manual	Year	Copy	Location	Registration Number
1	Agilent 7500 Series ICP-MS. Option Instruction Manual	2000	1	Metal Room, by ICP-MS	MM-9
2	Agilent 7500 Series ICP-MS. Application Handbook	2000	1	Metal Room, by ICP-MS	MM-10
3	Agilent 7500 Series ICP-MS. Hardware Manual	2001	1	Metal Room, by ICP-MS	MM-11
4	Agilent 7500 Series ICP-MS. ChemStation (G1834B) Operator's Manual	2001	1	Metal Room, by ICP-MS	MM-12
5	Agilent 7500 Series ICP-MS. Installation Guide	2002	1	Metal Room, by ICP-MS	MM-13
6	Agilent 7500 Series ICP-MS. Customer Maintenance Parts List	2002	1	Metal Room, by ICP-MS	MM-14
7	Agilent 7500 Series ICP-MS. Site Preparation Guide.	2003	1	Metal Room, by ICP-MS	MM-15
8	Agilent 7500 Series ICP-MS. Customer Maintenance Parts List	2004	1	Metal Room, by ICP-MS	MM-16
9	NESLAB Merlin Recirculating Chillers Thermo Manual	2005	1	Metal Room, by ICP-MS	MM-17
10	Operation Manual Microwave Digestion System MSP-1000	1996	1	Metal Digestion area	MM-18
11	HYDRA AA Automated Mercury Analyzer Manual, 150-00212 Rev. 1	2000	1 2	Mercury area QC Office	MM-19
12	iCAP 6000 Series ICP-OES Spectrometer	2007	1	Metal Room, by ICP-OES	MM-20
13	NESLAB Thermo Flex Recirculating Chillers. Installation. Operation. Basic Maintenance.	2007	1	Metal Room	MM-21

APPENDIX B

Wet Lab Instrument Manuals

#	Instrument Manual	Year	Copy	Location	Registration Number
1	DX-100 Ion Chromatograph Operator's Manual	1991	1	IC area	MW-1
2	Installation and Troubleshooting Guide for the ION PAC AG4A-SC Guard Column and AS4A-SC Analytical Column	1991/1992	1	IC area	MW-2
3	IBM PC Software Manual. UI20 Universal Interface Operator's Manual.	1994	1	IC area	MW-3
4	DX 100 Operational Techniques Maintenance and Troubleshooting	1996	1	IC area	MW-4
5	Peak net Software User's Guide Document #034914	1998	1	IC area	MW-5
6	Peak net Certificate of Software Validation, Release # 5.11	1999	1	IC area	MW-6
7	Installation and Troubleshooting Guide for the OMNIPAC PAX-100. Guard Column and Analytical Column.	1997/1998	1	IC area	MW-7
8	Appendix A- Principles of Conductivity Detection	1991	1	IC area	MW-8
9	Peak Net Software User's Guide Document #034914	1996	1	QC Office	MW-9
10	Konelab Reference Manual. Program version 5.0. Manual version A. Code 895341-4301.	2001	1	Konelab area	MW-10
11	NITON XLt 797Z. Alloy and Plastics Analyzer. User's Guide. Part Number 500-960 Version 4.2.	2004	1	XRF area	MW-11

APPENDIX C

Organic Lab. Instrument Manuals

#	Instrument Manual	Year	Copy	Location	Registration Number
1	Archon Purge and Trap Autosampler System	1997	1	VOA Lab	MO-1
2	O.I. Analytical 4552 w/s Autosampler	2001	1	VOA Lab	MO-2
3	Tekmar 3100 Purge and Trap Concentrator User Manual	1999	1	VOA Lab	MO-3
4	Tekmar LSC2000 User Manual	1991	1	VOA Lab	MO-4
5	Tekmar 3000 Purge and Trap Concentrator User Manual	1995	1	VOA Lab	MO-5
6	Teledyne Tekmar Velocity Manual (cd)	2006	1	VOA Lab	MO-6
7	Agilent 6890N Series Gas Chromatograph user info (cd)	2006	1	VOA Lab	MO-7
8	HP 5890 SeriesII and HP 5890 SeriesII PLUS Gas Chromatographs Manual	1993	1	VOA Lab	MO-8
9	5973N and 5973 inert Mass Selective Detector Hardware Installation	2003	1	VOA Lab	MO-9
10	HP 5971A Mass Selective Detector Hardware Manual	1989	1	VOA Lab	MO-10
11	HP 5972A Mass Selective Detector Hardware Manual	1993	1	VOA Lab	MO-11
12	HP 7673 Autosampler Users Manual	1987	1	Bookshelf by QA office	MO-12
13	EnviroQuant Users Guide	1993	1	Bookshelf by QA office	MO-13
14	Agilent 5973N inert MSD Hardware Installation	2003	1	Bookshelf by QA office	MO-14
15	Agilent 5973N inert MSD User Information cd	2003	1	Bookshelf by QA office	MO-15
16	Agilent 5975 Series Mass Selective Detector Hardware Installation	2005	1	Bookshelf by QA office	MO-16

17	Agilent 5975 Series Mass Selective Detector User Information (cd)	2005	1	Bookshelf by QA office	MO-17
18	Agilent 7683 Automatic Liquid Sampler User Information	2004	1	Bookshelf by QA office	MO-18
19	Agilent 7683 Automatic Liquid Sampler Controller Software Utility	2004	1	Bookshelf by QA office	MO-19

APPENDIX D
Grinding Instrument Manuals

#	Instrument Manual	Year	Copy	Location	Registration Number
1	Retsch SM 2000 High Performance Cutting Mill	1999	1	QA Office	MG-1
2	Turbula Shaker Mixer Type T2 F	1999	1	QA Office	MG-2
3	Retsch ZM 100 Ultra Centrifugal Mill	2002	1	QA Office	MG-3
4	Retsch DR 100 Vibratory Feeder	1999	1	QA Office	MG-4
5	Retsch ZM 100 Ultra Centrifugal Mill	1999	1	QA Office	MG-5

APPENDIX E
External Documents

#	Instrument Manual	Year	Copy	Location	Registration Number
1	2003 NELAC Standard	2003	1	QA Office	ED-1
2	International ANS/ISO/IEC 17025	2005	1	QA Office	ED-2
3	International Electrotechnical Commission IEC 623321 Ed. 1	2006	1	QA Office	ED-3

TITLE: PREVENTIVE ACTION

KEY WORDS: Preventive Action

COMMENTS: The SOP is converted to a new format, corrected editorial errors.
Italicized items indicate changes from the last revision.

REVISED BY: Brian Goyette February 28, 2008

APPROVALS:

QUALITY ASSURANCE MANAGER: Brian Goyette February 28, 2008
Brian Goyette

QUALITY ASSURANCE MANAGER: Lubitov February 28, 2008
Mary Lubitov

PREVENTIVE ACTION

1.0 SCOPE AND APPLICATION

1.1 Preventive action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

1.2 There are reviews in place to identify needed improvements and potential sources of non-conformities such as:

1.2.1 Management of Change

1.2.2 Internal Audits

1.2.3 Control Charting

1.2.4 Proficiency Testing

1.2.5 Validation of Data (by the supervisors)

1.2.6 Annual Management System Review

2.0 TERMINOLOGY

Preventive action: steps taken to prevent problems from occurring.

3.0 PROCEDURE

3.1 Management of Change is a process to manage change within the laboratory in a clear and thoughtful procedure ensuring proper items are addressed before any implementation of the changes. In general, the Management of Change procedure is divided into three main sections: Determination if change is needed, Plan to implement changes, and Implementation. For specific information on this procedure please refer to SOP # 276 Management of Change.

3.2 Internal audits are conducted in accordance with a predetermined schedule and procedure. All elements of the quality system including testing and operational activities should be audited at least annually. More frequent internal audits will depend on the critical nature of data, questionable results and audit findings. The schedule of the annual internal audits is prepared in the beginning of each year by the QA manager. The schedule might be subject to changes. In addition to the overall annual audits, audits covering particular laboratory activities might be conducted as needed (for example: auto pipette calibration, labeling standards and reagents, instrument maintenance, house keeping, purchasing and storage of disposable lab supplies and reagents, storage of laboratory records, samples and reagent disposal, review of contracts and tenders, handling of client complaints, etc.) LIMS is audited for assuring integrity and confidentiality of all data. For specific information on these procedures please refer to SOP # 246 Internal Audits.

3.3 Control charts are a means to demonstrate statistical control, and monitor the measurement process. In addition, they are useful in monitoring the stability of a particular analytical system. Through trends in the data, control charts alert the user to potential problems with the analytical system. Control charts for each method are developed to establish quality control acceptance criteria by using a minimum of twenty accuracy data points and twenty precision data points to constitute a database from which the control charts are constructed. The development of accuracy and precision control charts is described in SOP # 213 Control Charts.

3.4 Proficiency testing, EMT analyzes Proficiency Testing samples from a *designated Proficiency Testing Oversight Body (PTOB)/Proficiency Test Provider Accreditor (PTPA)-approved PT provider* two times per year for analytes required under the NELAC Standards. Wherever possible, EMT purchases samples from an ISO 9001 registered PT provider for analytes and matrices under ISO 17025-accreditation program and, where applicable, participates in the Proficiency Testing programs. The specific analytes and matrices analyzed are based on the current scope of the laboratory services. To maintain or obtain accreditation, EMT must successfully complete two PT studies at least 15 calendar days apart for each requested PT field of testing within the most recent three rounds attempted. For continuing accreditation, completion dates of successive proficiency rounds for a given PT field of testing shall be approximately six months apart. Failure to meet this semiannual schedule is regarded as a failed study.

3.5 Data validation/review by supervisors is a final check on the data as reported in the EMT LIMS. This review process includes spot checks on the raw data, and associated QC such as instrument calibration, and daily check standards. In addition to the QC checks, the data as reported in the LIMS, is checked to insure that computer calculations are correct, requested project analytes are reported, and required reporting limits are met. When this data *is* approved and validated by the supervisor it is then available for review and approval by the project management staff. For more information please refer to SOP # 234.

3.6 Annual Management System Review – At least once per year laboratory management conducts a review of the quality system to insure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review takes account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors. Every review, as well as the review findings and plans of action are documented. For more information please refer to SOP # 289.

4.0 REFERENCE

Laboratory Quality Assurance Manual, Environmental Monitoring and Technologies, Inc., 8100 North Austin Avenue, Morton Grove, IL 60053-3203.

Part B

Stat Analysis Corporation Standard Operating Procedures

TABLE OF CONTENTS

STAT Analysis Corporation

Standard Operating Procedure 005:

Document Control

Revision 01

Effective Date: March 15, 2005

Authors: Laurie Fetterman

Printed Name	Signature/Date
Dennis Jachim **Technical Manager**	_____
Laurie Fetterman **QA Manager/ Author**	_____
Laboratory Director	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

Copy Number: _____

Table of Contents

1.0 Identification of Test Method

SOP 005: Document Control

2.0 Applicable Matrix or Matrices

Not Applicable to this SOP.

3.0 Detection Limits

Not Applicable to this SOP.

4.0 Scope and Application

4.1 This SOP details the procedures for the control and maintenance of all SOPs (analytical and non-analytical), the Quality Manual, the Chemical Hygiene Plan, laboratory notebooks and various other documents, through a document control system that clearly indicate the time period during which the procedure or document was in force.

4.2 It is imperative to track and control all laboratory documents to assure that only the most current revision is being utilized within the laboratory. The document control system permits the retrieval of all-working files and archived records for inspection and verification purposes.

5.0 Summary of Test Method

This SOP details the assignment of unique laboratory ID numbers for both laboratory notebooks and SOP's. It also details the annual review of SOP's, documentation of updates and reviews, distribution, and archiving process of SOP's.

6.0 Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

Controlled Binder – A uniquely numbered binder that contains copies of specific SOPs or laboratory manuals. The contents of each controlled binder is updated and maintained by the QA Department. When laboratory documents are updated, a copy of the revised document is placed into the appropriate controlled binder(s) for laboratory use.

Controlled Copy – Controlled copies of laboratory documents are copies that are maintained and updated by the QA Department. These copies are assigned a unique control number and distributed throughout the laboratory for personnel to use.

Uncontrolled Copy – Uncontrolled copies of documents are not maintained nor updated by the QA department. At times, clients may request a copy of a STAT Analysis document for a QAPP or other purpose and is it classified as an uncontrolled copy.

7.0 Interferences

Not applicable to this SOP.

8.0 Safety

Not applicable to this SOP.

9.0 Equipment and Supplies

Not applicable to this SOP.

10.0 Reagents and Standards

Not applicable to this SOP.

11.0 Sample Collection, Preservation, Shipment and Storage

Not applicable to this SOP.

12.0 Quality Control

Not applicable to this SOP.

13.0 Calibration and Standardization

Not Applicable to this SOP.

14.0 Procedure

14.1 Document Identification

14.1.1 Each STAT Analysis Document, including SOPs (analytical and non-analytical), the Quality Manual, and the Chemical Hygiene Plan is assigned a unique identifier.

14.1.2 The SOP's are divided into numbered sections depending on their usage (See Appendix A). All SOPs, the applicable document number, revision number date of last review and their distribution are tracked in the STAT Analysis Document Master List located in \\Harrison\D\Quality Control\Laurie's TEMP\Tracking\Tracking-SOP. This table is updated as whenever a new SOP is required or an old SOP is retired. Obsolete SOP's are indicated in this table by marking the 'obsolete' column and entering the date collected.

14.1.3 All laboratory notebooks are assigned a unique lab book number. This number consists of a tracking number and a revision number. The QA Manager assigns the tracking number. The revision number starts with 1 and is increased by one, when a new revision is issued. The QA Manager tracks all notebooks numbers by the tracking number and the revision number.

NOTE: There are a few laboratory notebooks with the old numbering system still in use in the laboratory. The have been assigned a new logbook tracking number and

revision number. However, the analysts still use the old laboratory number when referencing these notebooks. This numbering system was designated by assigning the next consecutive number.

14.1.4 All forms are part of SOP's and they are tracked by the SOP system.

14.2 Annual Document Review and Updates

14.2.1 All STAT Analysis documents undergo an annual review from the date of last approval.

14.2.2 The QA Manager coordinates these annual reviews with the Laboratory Director and assigns a reviewer(s).

14.2.3 Documents are reviewed to current laboratory procedures and policies, current State and Federal regulations and to the most current approved method for compliance.

14.2.4 If a complete revision is needed:

14.2.4.1 Track Changes is turned on in Microsoft Word® to indicate new text by *italics* and deleted text by single line ~~cross-out~~. To permit ease of usage in the laboratory, the document will be printed without the above tracking displaying. However, all track changes are displayed on the screen and can be printed with changes highlighted if needed.

14.2.4.2 The SOP revision number will be incremented by 1 and a new effective date assigned. The procedure for assigning revision numbers and effective dates are detailed in QA100 SOP on SOPs.

14.2.5 If a complete revision is not needed, the SOP will be re-approved:

14.2.5.1 The documentation of this process will be a new signature page. The SOP is signed by the appropriate personnel with a statement placed on the signature page with an approval date directly below the effective date; "This SOP has been reviewed and it has been determined that no update is necessary at this time. Approved Date: _____".

14.2.5.2 The revision date and number remain unchanged.

14.2.6 The date of the last review is updated in the STAT Analysis Document Master List if changes were required or not.

14.2.7 Updates to SOPs or other documents between annual reviews:

14.2.7.1 Changes between annual reviews are made by an addendum to the document This addendum details the section of the document, a brief state of the change(s) and reason(s) for the change(s) to the SOP.

14.2.7.2 The QA department is responsible for completing addendum and placing it in the method binders. The addendum is placed at the back of the SOP. The Table of Contents is updated, by hand, to reflect the addition of the addendum.

14.2.7.3 The control copy number remains the same.

14.2.7.4 The SOP and supplementary addendums are archived . (See SOP 240 Archiving for details.)

14.2.8 When the document undergoes the annual review, the addendum *italicized* items are added to the document. The single line cross-out texts are removed.

14.2.9 Amendments by hand to any STAT Analysis Corporation documents are prohibited. Any changes to documents must be made by the following the procedures outlined in this SOP.

14.3 Document Control

14.3.1 Controlled copies of the SOPs, Quality Manual and other documents are located in each room in a controlled binder of the laboratory for personnel to use. The QA department assigns the binder numbers. Controlled copies of these documents cannot be reprinted in part or full. If additional copies are required in the laboratory, the QA Manager will issue another controlled copy.

14.3.2 As these documents are updated and revised, the QA department will replace each controlled copy with the revision in the controlled binders.

14.3.3 The original hardcopy document is archived by the QA department and all superceded controlled copies are destroyed (see QA SOP 240 Archiving). Original signatures identify it as the original document. The computer file is archived via the computer network and will include all the changes to the document (section 14.2.4).

15.0 Data Reduction, Calculations and Loading

Not applicable to this SOP.

16.0 Method Performance

Not applicable to this SOP.

17.0 Pollution Prevention

Not applicable to this SOP.

18.0 Data Assessment and Criteria for Quality Control Measures

Not applicable to this SOP.

19.0 Corrective Actions for Out-of-Control Data

Not applicable to this SOP.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

Not applicable to this SOP.

21.0 Waste Management

Not applicable to this SOP.

22.0 References

22.1 STAT Analysis Corporation Quality Assurance Manual

22.2 National Environmental Laboratory Accreditation Conference (NELAC), July 2002, Standards, USEPA Office of Research and Development, Washington, DC

22.3 SOP: 100 SOP on SOP's

22.4 SOP: 240 Archiving

23.0 Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data.

Appendix A – Section Categories of STAT Analysis Document Master List

Appendix A: Section Categories of STAT Analysis Document Master List

SOP ID	SOP Title
0000	General Corporation and Clerical
1000	General Lab Non-Analytical
2000	Miscellaneous Properties Test Methods
3000	Extraction, Preparation and Cleanup SOP's
4000	Analytical SOP's
5000	Asbestos Analytical SOP's
6000	Microbiology Analytical Methods

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 240

ARCHIVING

Revision 00
Effective Date: September 30 , 2002

Author: **Ian H. Graske**

Printed Name	Signature/Date
Dennis Jachim **Technical Manager**	_____
Ian H. Graske **QA Manager**	_____
Thomas M. Bauer **Laboratory Director**	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

Copy Number: _____

The absence of a Copy Number indicates this is an uncontrolled copy of the document supplied for information only.

Table of Contents

1. Identification of Test Method

SOP Title: Archiving

2. Applicable Matrix or Matrices

Not Applicable to the SOP

3. Detection Limits

Not Applicable to the SOP

4. Scope and Application

This SOP details the procedures used to archive and control the quality records, technical records, and the documents produced throughout all departments of STAT Analysis Corporation.

Archived records and documents are stored in a secure and readily retrievable manner. This is done to ensure that all records and documents that contain all pertinent information for historical reconstruction of the laboratory's testing related activities are complete and available for review.

Technical records include all testing related activities that encompass the laboratory's production of data related to individual test methodology, the ancillary support services, the sustaining records of sample condition, and the test reports submitted to the clients. The quality records include audit findings and reports, records of corrective actions, management reports and reviews, training records, reports from Performance Test (PT) sample vendors, client correspondence relating to specific projects or test activities, certificates and calibration reports from vendors (including sub-contractors), and any other records generated or received by the laboratory.

In addition to the technical and quality records, documents produced by STAT Analysis Corporation are also archived and stored in a secure and readily retrievable manner. Documents include the Quality Assurance Manual, Standard Operating Procedures (SOPs), the Personnel Manual, the Chemical Hygiene Plan, and any other documents that may relate to or affect the quality activities of the corporation.

Outdated test method references and related quality documentation obtained from outside sources are not archived. If these materials must be produced to supplement a laboratory record or document, the source of the reference method is contacted to procure the necessary documentation. An example would an outdated test method from a previous edition of EPA SW-846.

5. Summary of Test Method

Laboratory records are produced on a daily basis. The records are both handwritten original observations and electronic data files. Records are classified into several different groups based upon on type and the process that generated the record. Documents are produced on a periodic basis and are also classified into several different groups. Each type of record and document has its own time frame prior to collection and archiving into long-term storage. Once placed in long-term storage, records and documents are tracked by means of a filing system. They are accessed and controlled through the use of a sign-out sheet. Access is restricted to a limited number of corporation personnel. The stored records and documents are protected against loss, damage, and deterioration. After a minimum of five (5) years from last data entry for a record or archive date for a document, the records and documents are disposed of in a manner that ensures both

STAT Analysis Corporation's and its client's confidentiality. The minimum storage period may be increased dependent upon client request, regulatory requirement, or civil action order.

The QA Manager is responsible for ensuring that the procedures in this SOP are followed.

The procedures to be followed for archiving records and documents are outlined in Section 14 of this SOP. They include the following:

1. Identification
2. Collection
3. Indexing
4. Filing
5. Storage
6. Access
7. Maintenance
8. Disposal

The QA Manager, as part of the Internal QA Audit process, SOP 1220, also reviews this SOP to ensure that the requirements of this SOP are met and employees are in compliance. When deficiencies are noted by the QA Manager during his internal audit of this procedure, the corporation's corrective action process is employed to remedy these situations. The appropriate individual is tasked to take immediate corrective action and to implement the necessary changes in an expedient manner. Additional audits and follow up audits are performed as necessary to verify that corrective actions have been implemented and are successful in correcting the respective deficiencies.

Additional documentation is through the use of the Corrective Action Report (SOP 230 Corrective Action). Additional information relating to notebooks and records is found in SOP 1000 Control and Use of Laboratory Notebooks. Additional information relating to documents is found in SOP 005 Document Control.

This SOP is designed for the archiving and storage of hardcopy paper records.

Electronic records: Software used to produce records for data reduction, validation, reporting, archiving, and storage are addressed in SOP 1500 Computer Network.

6. Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

Not Applicable to the SOP

8. Safety

Filled Bankers Boxes can be heavy. Use caution when moving and lifting these boxes.

9. Equipment and Supplies

Not Applicable to the SOP

10. Reagents and Standards

Not Applicable to the SOP

11. Sample Collection, Preservation, Shipment and Storage

Not Applicable to the SOP

12. Quality Control

Not Applicable to the SOP

13. Calibration and Standardization

Not Applicable to the SOP

14. Procedure

14.1 Identification of Laboratory Records and Documents

Laboratory records and documents are classified into several categories. Additional information about records may be found in SOP 1220 Internal Quality Assurance Audit Section 14.8.

14.1.1 Test Report File: Report, Chain-of-Custody, Case Narrative, supporting Quality Control Report and client correspondence relating to specific projects or test activities.

14.1.2 Notebooks and customized forms are defined in SOP 1000 Control and Use of Laboratory Notebooks.

14.1.3 Instrument generated test data hardcopy records

14.1.4 Internal audits and external audits: findings and reports

14.1.5 Corrective actions

14.1.6 Management reports and reviews

14.1.7 Training records, including the employee Signatures and Initials List

14.1.8 Reports from PT providers

14.1.9 Certificates and calibration reports from vendors (including sub-contractors)

14.1.10 Documents include: The Quality Assurance Manual; Standard Operating Procedures (SOPs): Both technical and administrative are listed on the SOP Document Control Master List, SOP 005, the Chemical Hygiene Plan, and any other documents that may relate to or affect the quality activities of the corporation.

14.1.11 Outdated test method references and related quality documentation obtained from outside sources are not archived.

14.2 Collection of Laboratory Records and Documents

Records and documents no longer needed for quick reference or to produce a test report are filed in temporary storage. Those employees with access to these temporary storage areas include the President, QA Manager, Technical Manager, Laboratory Director, Department Managers, and Project Managers. Temporary storage provides easy access to the material. A formal sign-out sheet to track the movement of these records and documents is not necessary with the exception of test report files. After the end of the temporary storage period, the records and documents are collected by the QA Manager, with the help of the Technical Director and the Project Managers, and placed in long-term storage. The temporary storage areas and residence time are as follows:

14.2.1. The test report file is placed in a locked filing cabinet that has defined limited access. The reports may remain in the locked cabinet for up to one year prior to archiving.

14.2.2. Completed laboratory notebooks and instrument generated test data may remain in the respective department for up to two months prior to archiving.

14.2.3. Completed forms may remain in a controlled active access folder for up to one year prior to archiving.

14.2.4. Audit findings and reports may remain in a controlled active access folder for up to two years prior to archiving.

14.2.5. Corrective actions reports may remain in a controlled active access folder for up to two years prior to archiving.

14.2.6. Management reports and reviews may remain in a controlled active access folder for up to one year prior to archiving.

14.2.7. Training forms, for employees no longer working for the corporation, may remain in a controlled active access folder for up to six months prior to archiving.

14.2.8. Reports from PT providers may remain in a controlled active access folder for up to two years prior to archiving.

14.2.9. Client correspondence relating to specific projects or test activities may remain in a controlled active access folder for up to one year prior to archiving.

14.2.10. Certificates and calibration reports from vendors (including sub-contractors) may remain in a controlled active access folder for up to one year prior to archiving.

14.2.11. Documents may remain in a controlled active access folder for up to one year prior to archiving.

14.3 Indexing of Laboratory Records and Documents

The following is a responsibility of the QA Manager.

14.3.1. The QA Manager maintains and controls a master list of all record types and documents produced by the corporation (see Attachments 1 through 12; for notebook master list see SOP 1000). This list includes the record or document control number, title or type, department or area location, effective dates, collection date (archive date into long-term storage), projected date for disposal, actual disposal date, and initials.

14.4 Filing of Laboratory Records and Documents

The following is a responsibility of the QA Manager, Department Managers and the Project Managers:

14.4.1 Records and documents are removed from their temporary storage locations.

14.4.2 Records and documents are filed in "Bankers Box" cardboard storage boxes.

14.4.3 Using indelible ink or a typed label, each box is clearly labeled (on both ends) with the following information: Unique Box ID number, type of record or document; instrument number for test data, if applicable; effective dates (beginning and ending date), work order numbers (beginning and ending), if applicable, filing date, initials of filer, and projected disposal date.

14.4.4 The contents label must be easily readable (perhaps from some distance if the boxes are stacked on tall shelves) to ensure efficient retrieval of requested records.

14.4.5 Records may be divided and placed in labeled manila folders for easier access. This is normally used for instrument batch data packets.

14.4.6 A summary sheet, with additional details for easy reference, may also be placed in the box.

14.4.7 The boxes are filled to an appropriate level and then closed.

14.5 Storage of Laboratory Records and Documents

The following is a responsibility of the QA Manager.

14.5.1. The filled boxes are placed in storage.

14.5.2. Storage areas are defined as the following areas: QA Manager's office, Customer Service Manager's office, Laboratory Director's office, Archived Records Storage Area and an offsite storage location.

14.5.3. Storage areas are held secure under lock and key.

14.5.4. Access to the storage areas is restricted to the following laboratory personnel: The President, QA Manager, Technical Manager, Laboratory Director, Customer Service Manager, Department Managers, and Project Managers.

14.5.5. Storage areas must provide adequate security to ensure against loss, damage, and deterioration. Records and documents are not exposed to water or laboratory chemicals that could jeopardize their condition.

14.5.6. Boxes are not placed directly on the floor. They are arranged in a neat, orderly fashion on sturdy shelves.

14.5.7. The boxes are stored such that similar records are in the same location of the storage area.

14.5.8. The boxes must be stored such that the contents label is easily readable to ensure efficient retrieval of requested records. Boxes are not to be stacked more than three high.

14.6 Access to Laboratory Records and Documents

The following is a responsibility of the QA Manager and the Technical Manager.

14.6.1 Access to archived records is restricted to the following laboratory personnel: The President, QA Manager, Technical Manager, Laboratory Director, Customer Service Manager, Department Managers, and Project Managers.

14.6.2 Access to archived records is maintained through the use of a sign-out sheet (see Attachment 13).

14.6.3 The sign-out sheet contains the following information: Box ID number, type of record or document; date(s) of record, work order number (for test reports), if applicable, date removed from storage, initials of person removing the record. In addition, the person returning the record must initial and date the sign-out sheet in the appropriate area. The person returning the record should then place it back in the appropriate box.

14.6.4 Archived records may be transported from one department to another for information or photocopying purposes. They should remain in the possession and control of the person who removed the records from storage. It is not permitted to remove archived records from the building unless special permission is granted from top management.

14.7 Maintenance of Laboratory Records and Documents

The following is a responsibility of the QA Manager, Laboratory Director, Technical Manager and President:

14.7.1 The storage areas are maintained under lock and key.

14.7.2 Environmental conditions are maintained to prevent loss, deterioration, or damage to the archived records.

14.7.3 The QA Manager, as part of the Internal QA Audit process SOP 1220, reviews the storage area, storage boxes, and sign-out sheet to ensure compliance with the SOP.

14.7.4 In the event that the corporation transfers ownership, the new proprietors retain sole custody and complete responsibility for all records pertaining to client samples. Clients will be notified in writing of the ownership transfer.

14.7.5 In the event that the corporation ceases business operations, management will make arrangement for the safety and security of the records until the projected disposal date. Clients will be notified in writing of these arrangements and the procedure to access their data, if necessary.

14.7.6 In case of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

14.8 Disposal of Laboratory Records and Documents

The following is a responsibility of the QA Manager.

14.8.1 After a minimum of five (5) years from last data entry for a record or archive date for a document, the records and documents are disposed of in a manner that ensures both STAT Analysis Corporation's and its client's confidentiality. **Records pertinent to the National Lead Laboratory Accreditation Program (NLLAP) are maintained for a period of at least ten (10) years.** All records are shredded and sent to a paper recycler.

14.8.2 The minimum storage period may be increased dependent upon client request, regulatory requirement, or civil action order.

14.8.3 The QA Manager, or his assignee, removes the archived record from the long-term storage area after the projected disposal date listed on the storage box. After the box is disposed, the QA Manager records the actual disposal date on the appropriate master records list.

15. Data Reduction, Calculations and Loading

Not Applicable to the SOP

16. Method Performance

Not Applicable to the SOP

17. Pollution Prevention

Records disposal is done in an environmentally friendly manner.

18. Data Assessment and Criteria for Quality Control Measures

Not Applicable to the SOP

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the Internal QA Audit review findings determine that the laboratory performance for records archiving and storage is judged to be out of control, the problem must be immediately identified and corrected. If it is determined through the corrective action process that client data may have been lost or damaged, the time frame for the out of control situation will be determined. This will identify what client samples were analyzed during the time frame. Immediate corrective

action includes written notification to clients that the data produced by the laboratory may not be retrievable and defensible.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable procedures or potentially lost data are revealed through the Internal QA Audit review process, the first option is to identify the problem and determine its extent.

The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of this potentially adverse situation must be approved and recorded by the Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21. Waste Management

Not Applicable to the SOP

22. References

22.1 SOP 100 SOP on SOPs
22.2 SOP 230 Corrective Actions
22.3 SOP 005 Document Control
22.4 SOP 1000 Control and Use of Laboratory Notebooks
22.5 SOP 1220 Internal Quality Assurance Audit
22.6 SOP 1500 Computer Network
22.7 STAT Analysis Corporation Quality Assurance Manual

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

The forms used for the Archived Records Master List are attached.

ATTACHMENT 1: ARCHIVED NOTEBOOK MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 2: ARCHIVED DOCUMENTS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 3: ARCHIVED SOPs MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 4: ARCHIVED FORMS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 5: ARCHIVED QUALITY REPORTS AND AUDIT REPORTS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 6: ARCHIVED CORRECTIVE ACTIONS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 7: ARCHIVED TRAINING RECORDS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 8: ARCHIVED INSTRUMENT DATA MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 9: ARCHIVED TEST REPORTS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 10: ARCHIVED PT REPORTS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 11: ARCHIVED CERTIFICATES, CALIBRATION REPORTS MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 12: ARCHIVED CLIENT CORRESPONDENCE AND QAPPs MASTER LIST

Control #	Type	Dept	Effective Dates	Archived	Disposal projected	Disposal actual	Initials

NOTE: Enter the following information into the table: record or document control number, title or type, department or area location, effective dates of use, collection date (archive date or date placed in long-term storage), projected date for disposal, actual disposal date, initials to indicate disposal was performed.

ATTACHMENT 13: ARCHIVED RECORDS SIGN-OUT SHEET

Box #	Type	Date of Record	Control # Work Order #	Date Removed	Initials	Date returned	Initials

NOTE: Enter the following information into the table: Box ID number, type of record or document; date(s) of record, record control number or work order number (for test reports), if applicable, date removed from storage, initials of person removing the record. In addition, the person returning the record must initial and date the sign-out sheet in the appropriate area.

SOP ADDENDUM

SOP TITLE: SOP 240 Archiving, Revision 00
Effective Date: September 30, 2002

Issued by Ian Graske, QA Manager 1/17/03

Approved by: Dennis Jachim, Technical Manager 1/17/03

Date of Issue: January 17, 2003

The following information is added to the SOP:

Section 4.8.1 (edit this section, additions are *italicized*)

14.8.1 After a minimum of five (5) years from last data entry for a record or archive date for a document, the records and documents are disposed of in a manner that ensures both STAT Analysis Corporation's and its client's confidentiality. *Records pertinent to the National Lead Laboratory Accreditation Program (NLLAP) are maintained for a period of at least ten (10) years.* All records are shredded and sent to a paper recycler.

END OF ADDENDUM

The initials/signature of the QA Manager and the Technical Manager indicate that this is a controlled document.

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 300

Sample Receiving and Login Procedures

Revision 00
Effective Date: September 18, 2002

Author: **Ian H. Graske**

Printed Name	Signature/Date
Craig Chawla **Customer Service Manager**	_____
Dennis Jachim **Technical Manager**	_____
Ian H. Graske **QA Manager**	_____
Thomas M. Bauer **Laboratory Director**	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

Copy Number: _____

The absence of a Copy Number indicates this is an uncontrolled copy of the document supplied for information only.

Table of Contents

1. Identification of Test Method

SOP Title: Sample Receiving and Login Procedures

2. Applicable Matrix or Matrices

Not Applicable to the SOP

3. Detection Limits

Not Applicable to the SOP

4. Scope and Application

This SOP includes procedures that are intended to document sample possession during each stage of sample handling from receipt of the laboratory up to distribution of the sample to the appropriate laboratory department. The purpose of these procedures is to provide accountability for and documentation of sample integrity from the time samples are collected until sample disposal.

5. Summary of Test Method

Chain-of-custody procedures are a necessary element in a program to assure one's ability to support data and conclusions adequately in a regulatory situation, but custody documentation alone is not sufficient. A complete data defensibility scheme should be followed. This SOP addresses chain-of-custody procedures as they relate to the laboratory handling of samples. It also addresses the login procedures used to enter the sample identification into the LIMS. The LIMS provides a permanent record of the receipt of all sample containers that are logged into the LIMS. Samples not logged in, due to improper sample condition and subsequent are not tracked through LIMS. Samples for specific projects or analyses may be reported in a dedicated logbook for tracking purposes (i.e. samples for lead analysis for abatement for industrial hygiene purposes).

The sample acceptance policy is as follows:

- Proper, full and complete documentation, including the sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample.
- Unique identification of samples using durable labels completed in indelible ink.
- Appropriate sample containers are used with appropriate preservation.
- Adequate sample volume has been collected.
- Samples are received within appropriate holding times.
- Samples for volatiles analyses do not contain headspace.

Data from samples that do not meet the sample acceptance criteria will be unambiguously flagged to define the nature of the variance.

Appropriate procedures are taken when samples show signs of damage or contamination.

6. Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

Not Applicable to the SOP

8. Safety

8.1 Proper protective equipment must be worn. At minimum, this consists of gloves, a lab coat and safety glasses.

8.2 All shipping containers (coolers) are opened in an adequately ventilated area to assure worker safety.

8.3 When possible, determine the source of the samples and any special hazards that might be associated with them.

8.4 Some samples, when sealed in containers will build up pressure. Samples that indicate pressure should be brought to the attention of the Safety Officer or Laboratory Management.

8.5 There will be no eating, drinking, or smoking in the sample receiving area.

9. Equipment and Supplies

9.1 Calibrated Digital Thermometer

9.2 pH paper – wide range

9.3 Potassium Iodide starch paper

9.4 Repipet Dispensers – to add preservatives.

10. Reagents and Standards

10.1 Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Standard and Reagent Preparation. Analytical reagent grade chemicals are used for all preservatives.

10.2 Preservatives

10.2.1 Nitric Acid, concentrated
10.2.2 Nitric Acid, 1:1
10.2.3 Hydrochloric Acid, 1:1
10.2.4 Sulfuric Acid, 1:1
10.2.5 Sodium Hydroxide, 10N
10.2.6 Sodium Thiosulfate, granular or powder
10.2.7 Ascorbic Acid, granular or powder
10.2.8 Zinc Acetate

11. Sample Collection, Preservation, Shipment and Storage

Sample bottles and coolers are prepared in the laboratory prior to shipment to the collection site or for delivery to clients. A list of requested test parameters or bottle order request is received at the laboratory. The bottle prep person uses the Sample Containers List (see Attachment 3) as a reference to assemble suitable containers to satisfy the submitted bottle order. Appropriate preservatives are added to the applicable bottles prior to shipment. The Bottle Order Form (see Attachment 4) is used to record the number and type of containers in the shipment. Coolers, containing dedicated temperature blank bottles, are also provided in the shipment.

Samples are shipped to the laboratory via commercial courier, client courier, or STAT laboratory courier. If samples arrive during normal business hours, samples are received by the sample custodian or a designated alternate. Samples that arrive after normal business hours will be secured in the sample custody room (currently room 315) or remain in the custody of the building security guard. The sample custodian will receive the samples the next business day.

12. Quality Control

Not Applicable to the SOP

13. Calibration and Standardization

Not applicable to the SOP

14. Procedure

14.1 While wearing proper protective equipment, (a minimum of gloves, a lab coat, and safety glasses) all shipping containers (coolers) are opened in an adequately ventilated area to assure worker safety.

14.2 The sample custodian records all information concerning sample condition on the Chain-of-Custody (COC Attachment 1) and the Sample Receipt Checklist Form (Attachment 2). Information

on the COC should include, but is not limited to, the following items: sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample. If there is any missing information on the COC, the client is notified and the information is requested at that time. If no COC is received with the samples, the Project Manager is immediately notified by the sample custodian to contact the client and take appropriate corrective action. A Sample Receipt Checklist Form is completed for each cooler, box or group of hand delivered samples to note type of package, shipping mode, temperature and other information pertaining to the condition of the samples upon receipt.

14.3 All shipping containers (coolers) are examined to verify that the custody seal is intact (if present). The parts of the custody seal are maintained in the client folder after opening. The shipping containers (usually coolers) are received, opened and prioritized according to holding time and turn-around (Emergency, Rush or Standard) requirements. The Chain-of-Custody is signed and dated (including time) at the time of sample receipt by the sample custodian.

14.4 The temperature of the shipping cooler and/or temperature blank are measured to determine if proper temperature has been maintained. Samples that have been received within six hours of collection and on ice will be noted as being received "On Ice" as complete cooling to 4°C will not have been completed by that time.

14.5 Temperature Check Procedures:

14.5.1 Cooler temperatures should be within 0.1-6.0°C. The temperature of a cooler must be measured as soon as possible after it is opened.

14.5.2 The two (2) methods to check temperature are using a temperature blank and taking a representative cooler temperature. The temperature must be measured using a temperature blank, if available, before using the representative cooler temperature procedure. The temperature is measured using a calibrated digital thermometer. Check the thermometer tag prior to use to ensure that the thermometer is still within the calibration time period.

14.5.3 If a cooler contains a temperature blank, a digital probe thermometer is used to measure the temperature. Remove the cap of the temperature blank and insert the probe. Allow the temperature reading to stabilize and then record it.

14.5.4 If a cooler does not contain a temperature blank, a digital probe thermometer is used to measure the representative cooler temperature. The digital probe is inserted in the cooler in a spot that is representative of where the samples reside in the cooler. If the samples are immersed in wet ice, the probe is put in the ice. If the cooler contains blue ice packs, the probe is placed next to a sample bottle. The cooler lid is closed. Allow the temperature reading to stabilize and then record it.

14.6 The temperature is immediately recorded on the COC. It is also recorded on the Sample Receipt Checklist Form during the log in procedures in LIMS.

14.7 The condition of the jars (leaking, broken, mislabeled or unclearly labeled) is checked. Exceptions are noted on the Sample Receipt Checklist Form and the client is notified of the impact that the exception will have on the quality of data generated. Data from samples that do not meet the sample acceptance criteria will be unambiguously flagged to define the nature of the variance.

14.8 The samples are examined and compared with the Chain-of-Custody and any other documentation received with the samples. Additional documentation may indicate that the sample is to be analyzed as received or that other preparation must be first performed prior to analysis. An example would be the compositing of samples from multiple sample containers into one composite sample prior to testing. The COC is examined for accuracy and completeness. For all samples, especially environmental or industrial hygiene samples, it is vital that all COC procedures are followed properly due to the potential for litigation. All environmental samples delivered to the lab should be accompanied by a COC. This is necessary to preserve the security of samples as evidence. Samples are considered secure for evidentiary purposes if they are in your possession, within view, or in a secured area. The laboratory is considered secure because access is limited and monitored 24 hours a day by security personnel. The COC record is used to document the change in possession from sampling, delivery, and receipt by the laboratory. Any problems associated with samples on the COC are immediately noted on a Sample Receipt Checklist Form. The name of the assigned STAT Project Manager is also recorded on the Sample Receipt Checklist Form. The Project Manager is immediately notified of the problem(s) and is responsible for communicating with the client on how to resolve issues associated with the problem samples.

14.9 Samples received and sample jar labels are compared against those listed on COC. Samples, and the requested test analyses, are compared to the sample requirements for those tests (Containers, Preservatives, Holding Times listed on Attachment 3). These observations are recorded on the Sample Receipt Checklist Form. Sample holding times are verified for sample acceptance. The client is immediately notified if holding times have been exceeded.

14.10 Sample pH is verified for those samples that require specific chemical preservation. The sample pH result is recorded on the Sample Receipt Checklist Form. VOA water samples are not checked for pH at time of receipt but are checked after analysis. VOA samples are checked for headspace at time of receipt. Samples for cyanide analysis are checked for free chlorine at time of receipt. The sample free chlorine result is recorded on the Sample Receipt Checklist Form. Samples that require additional preservation for pH adjustment or require the removal of free chlorine are treated by the sample custodian. The identification and amount of chemical preservative is recorded on the Sample Receipt Checklist Form. The client is immediately notified if samples have not been properly preserved.

14.10.1 Sample pH check: Shake the sample bottle, set the sample bottle on the counter and remove the cap. Carefully place a small drop of liquid from the cap onto the wide range pH paper. Note the pH. If all samples are properly preserved, note as such on the Sample Receipt Checklist Form. If the pH is not appropriate, dispense 1 to 2 ml of the proper chemical preservative (acid or base) into the sample bottle. Cap the bottle, shake the bottle, and repeat the pH test to ensure proper pH adjustment. Do not add more than 5 mL preservative per liter sample volume. Note the sample ID, preservative amount and ID, and any applicable sample observations (if applicable) to the Sample Receipt Checklist Form.

14.10.2 Chlorine check: Shake the sample bottle, set the sample bottle on the counter and remove the cap. Carefully place a small drop of liquid from the cap onto the Potassium Iodide starch paper. Note any color change. The paper will turn blue if chlorine is present. If all samples are negative for chlorine, note as such on the Sample Receipt Checklist Form. If chlorine is present, dispense in a small amount of Ascorbic acid (approximately 600 mg) for cyanide samples only or Sodium thiosulfate (approximately 100 mg) for other analyses into the sample bottle. Cap the bottle, shake the bottle, and repeat the chlorine test to ensure proper chlorine neutralization. Do not add more than 500 mg Sodium Thiosulfate per liter sample volume. Note the sample ID, preservative amount and ID, and any applicable sample observations (if applicable) to the Sample Receipt Checklist Form.

14.11 The Chain of Custody, Sample Receipt Checklist Form, and Waybill are placed in a Job Folder, which is labeled by STAT Work Order Number and Client Name. All information/analytical reports pertaining to the specific job are stored in this folder. This includes quotes, faxes, correspondences, analytical reports, sub-contracted analytical reports, etc.

14.12 The samples are set out on the counter according to the Chain-of Custody. The Project Manager is notified of any problems or discrepancies (such as broken sample jars, insufficient sample amount to perform analysis, etc) as soon as possible and contacts the client, if warranted. Problems or discrepancies must be noted on the Sample Receipt Checklist by the person setting out the samples and the resolution must be noted by the Project Manager before proceeding. The resolution is one of the following:

1) Samples are rejected, labeled for disposal, and no further analysis is performed on the samples.
2) Client states to proceed with the analysis, the data is flagged accordingly to indicate the criteria that did not meet the sample acceptance policy.

Any correspondence between the laboratory and the client is documented.

14.13 Samples requiring refrigeration are stored in the appropriate sample refrigerators. Samples not requiring refrigeration are placed in the appropriate department storage areas.

14.14 To minimize the time a sample is kept on the counter, any samples not able to be logged in immediately due to problems or discrepancies, should be moved to refrigerated storage until there is resolution.

14.15 Samples received by the laboratory do not come from mixed waste sites. Thus, samples are not routinely screened for radioactivity. If a client wants to submit potentially radioactive samples to the laboratory, the client must make arrangements to pre-screen these samples in the field and record, on the COC, that the level of radioactivity of these samples is not harmful.

14.16 Sample information is entered into the computer from the Chain-of-Custody Record. Information on the Chain-of-Custody must agree identically with the information entered into the LIMS. If a

Project Manager wants information to be entered differently than stated on the Chain-of-Custody, each Chain-of-Custody must be fully edited (dated and initialed) to state so by the Project Manager.

14.17 Each group of samples (or job) is assigned a unique work order number by the LIMS program. The work order number is a seven-digit sequentially assigned number. The first two digits represent the year, the second two digits represent the month, and the last three digits represent the sequential receipt of that particular group of samples. Thus work order number 0208050 would include the sample group submission that occurred in year 2002, month of August, and the fiftieth sample group (job) submitted in that month. The next job received would be 0208051, etc. The first job received in the next month, September 2002, would be number 0209001.

14.18 The LIMS is used to generate the sample log and assign sample numbers that are an unequivocal link to the sample field identification code or name. The sample log generates a unique work order for a specific project or group of samples. All sample containers are labeled with a unique laboratory sample number. This numbering system is also used to uniquely identify separate containers of the same sample submitted within the work group. The unique laboratory sample number is used throughout all of the laboratory records to identify the sample and any subsequent sub-samples, extracts, or digestates of the original sample. The entry of sample information into the LIMS is password controlled. Thus, the name of the person entering the information is recorded. The following information is entered into the sample log:

14.19 Procedure for logging samples into the LIMS is as follows:

14.19.1 Double click on "STATMDE".
14.19.2 Login in to the system.
14.19.3 Click on "Work Orders" button
14.19.4 To start a new work order, place cursor in the "work order" field and click on "Add" button. Document the work order number on the COC in the "work order No:" field. If it is not a STAT Analysis COC, write the work order number in the comments section.
14.19.5 Choose client in "Client ID" field.
14.19.6 Select the appropriate client contact in the "Client ID" field.
14.19.7 Enter the client project number and name in the "order" field. This information should be on the COC.
14.19.8 Enter the client's project location in the "Location" field.
14.19.9 Enter the date received in the "Received" field.
14.19.10 Enter the required turnaround time in the "TAT" field.
14.19.11 The project due date automatically appears in the "Date Due" field.
14.19.12 Click on "Report Options" button to verify that the client contact invoicing information is correct as well as the reporting format.
14.19.13 Click on the "Login" button to proceed.
14.19.14 Enter the client's sample identification name or number in the "Client Sample ID" field.
14.19.15 Enter in any additional sample information (depth, etc.) in the "Tag Number" field.
14.19.16 Enter in the date and time collected (mm/dd/yy followed by one space and then the time of day in "military time") in the "Collection Date" field.
14.19.17 Choose the appropriate matrix in the "Matrix" field.

14.19.18 Choose the appropriate container type in "Bottle Type" field. If multiple analysis are requested for each sample, start with organics then move on to inorganics. Example: VOC's, SVOC's, Pest/PCB's, Metals.

14.19.19 Enter in the number of sample jars in the "# Containers" field.

14.19.20 Enter in the location of the sample container in the "Storage/Status" field. Example: "Room 326" or "refrigerator 7".

14.19.21 Enter in the COC number in the "COC ID" field.

14.19.22 Enter in the appropriate analysis requested by the client in the "Test Group" or "Test" field.

14.19.23 You are now done logging in that sample jar.

14.19.24 If there are multiple bottle types for each sample bottle distinguished by preservative, click on "Add Frac". By doing this each sample container receives it's own unique identification number.

14.19.25 Information from the previous sample is copied onto this fraction. Be sure to change the information as necessary.

14.19.26 At this point, each sample container has a unique lab number. Each jar will have a printed durable label that has the following information: Work Order Number, Sample ID Number, preservation fraction, and the bottle number of total number of bottles with each particular preservation (i.e. 0207113-001A bottle 1 of 3).

14.19.27 If there are multiple samples on the COC for the same analysis, click on the "Multi-log" button, enter the information as on the COC, click on the "post" button to assign STAT Analysis sample ID numbers.

14.19.28 If there are additional samples that have different requested analysis, click on "ADDSamp" button. Then follow the procedure starting from section 14.19.3 when you enter in the client sample ID.

14.19.29 You can copy requested test analysis by clicking on the "CopySamp" button and selecting the sample number that has the identical requested analysis as the sample you are now logging in.

14.19.30 Click on "Show Datasheet View" button to conduct a preliminary review of the workorder. Verify that the information is correct. This information includes sample ID number, work order number, date received, client sample ID number, collection date, etc.

14.19.31 When sample log in is complete, click on the "labels" button.

14.19.32 Using the arrow buttons on the "sample selection box", move the desired sample id's from "Available" to "Selected".

14.19.33 Click on the "OK" button.

14.19.34 The labels will appear on the print preview screen.

14.19.35 If labels are correct, click on the printer icon to print.

14.19.36 Once printed, attach the durable labels to the appropriate jars.

14.19.37 When all the log in and labeling procedures are completed, then distribute sample jars to the appropriate sample storage locations.

14.19.38 Click on "InvoiceInfo" button to verify that the billing information is correct. Surcharges, discounts and PO numbers can be entered into the appropriate fields.

14.20 The Sample Receipt Checklist Form must be completed after all samples in the work order are logged into LIMS.

14.20.1 Click on the "ChkList" button.
14.20.2 Place the cursor in the "received by" field and enter in the correct personnel.
14.20.3 Enter in the method of receipt in the "carrier name" field.
14.20.4 Read the thirteen questions and click on the appropriate answer (Yes, No or Not Present).
14.20.5 Enter in the temperature received in the "temperature" field as °C.
14.20.6 When completed, click on the "preview" button to view the completed form.
14.20.7 If the form is acceptable, click on the print icon. If it is not acceptable, close the print preview screen and make the necessary changes. If it is now acceptable, you may either click on the "generate" button to print or click on the "preview" button to view again.
14.20.8 After printing the Sample Receipt Checklist, the person who logged in the samples must sign and date the form.
14.20.9 If "No" is the answer for any of the questions, a description must be written in the "Comments" section of the printed form. If a discrepancy or a deficiency warrants a corrective action, a formal corrective action is initiated (see SOP 230 Corrective Action) and it is noted in the "Corrective Action" portion of the printed form.
14.20.10 The Sample Receipt Checklist is then placed in the work order folder along with the COC, the Waybill, and any other documentation or paperwork received with the samples. The work order folder is then given to a Project Manager for review. If the COC and Sample Receipt Checklist are acceptable, the reviewer then initials and dates the form.
14.20.11 In addition to a review of the COC and the Sample Receipt Checklist, the Project Manager performs a secondary review of the information entered into the LIMS. This may be done electronically or the LIMS work order sheets may be printed for review. If the review is satisfactory, the Project Manager initials and dates the Sample Receipt Checklist form. If the review is not satisfactory, the Project Manager notes the deficiencies and returns the work folder to the login person for corrections.
14.20.12 If there are any comments on the Sample Receipt Checklist form, the Project Manager then addresses the discrepancies or deficiencies and notifies the client immediately. The following portions of the form are then completed: Client Contacted, Date Contacted, Person Contacted, Contacted by, and regarding any additional correspondence between the laboratory and the client is documented and placed in this folder.

14.21 All samples must be entered into the LIMS and labeled before being distributed in the laboratory. An exception may occur for a priority sample or a sample near holding time expiration. A temporary lab ID number may be assigned to the sample and recorded on the COC. The laboratory records will contain this number and the subsequently assigned LIMS number.

14.22 Occasionally, samples are received for analyses that are not performed at STAT Analysis and are subcontracted to another laboratory. The samples are received and logged in according to the procedures outlined in this SOP. For sample submission procedure to another laboratory, see Customer Service SOP 220.

14.23 Samples to be analyzed at the lab are placed in their designated storage locations. Samples are stored away from standards, reagents, food, and other possible contaminants. Highly contaminated

samples may be stored in a fume hood or in a separate storage cooler on ice if refrigeration is required. NOTE: Volatile samples are to be placed in the Volatiles refrigerator to eliminate cross-contamination from other departments that use solvents in their extraction process.

14.24 Worksheet Folders

14.24.1 The Chain-of-Custody, the Sample Receipt Checklist (generated from LIMS) and all other documentation associated with the samples are placed in the Work Order folder.

14.24.2 The Sample Receipt Checklist is signed and dated to reflect who logged in the samples.

14.24.3 The folders are distributed to the Project Manager for review. The Project Manager performs the review of the login either by reviewing paper copies of the requested tests for the samples or by reviewing the LIMS directly. If corrections are not required, the Project Manager will initial the Cover Sheet with date approved and reviewed and, after processing the paperwork, will return the folders to the sample handling department for filing. The Sample Receipt Checklist is initialed and dated after review.

14.24.4 If corrections are required, the project manager will annotate the corrections to be made on the Cover Sheet and return the folder to the sample handling department. The sample custodian will immediately correct the information in the computer and regenerate the affected documents. The corrected folder will be returned to the Project Manager for review. The Project Manager will initial the Cover Sheet with the date reviewed and approved and, after processing the paperwork, will send the folder to the sample handling department for filing.

14.24.5 If a client upgrades the priority to either emergency or rush turn-around, the change is written on the Chain-of-Custody Record by the laboratory contact who received the request. The Chain-of-Custody Record is also annotated with the name of the person who requested the upgrade, the date the request was made, and the laboratory contact. Those changes are made in LIMS and the appropriate Department Manager is verbally notified by the Project Manager of the new priority.

14.24.6 Active, approved files are maintained in the sample handling department sequentially by order number.

15. Data Reduction, Calculations and Loading

Not Applicable to the SOP

16. Method Performance

Not Applicable to the SOP

17. Pollution Prevention

Not Applicable to the SOP

18. Data Assessment and Criteria for Quality Control Measures

Not Applicable to the SOP

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action. Corrective action, if necessary, will be implemented by the Project Manager.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director will review data and discusses options with the client. Re-analysis or reporting the data with qualifications are alternatives. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.

20.1 The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

20.2 Client Requested Modifications:

20.2.1 Clients must request modifications from the laboratory SOP in writing to the lab.

20.2.2 The Laboratory Director, Technical Manager and Quality Assurance Manager will evaluate the requested client deviations, determine the feasibly of the deviation and the potential effects on the data.

20.2.3 If it is determined that the laboratory will perform the requested deviations, the Laboratory Director, Technical Manager and Quality Assurance Manager will decide if a method validation study is required.

20.2.4 The designated project manager will retain all documentation concerning the requested deviation, including all correspondence with the client, in the client folder.

20.2.5 The final analytical report must include the statement "*This report has analyses performed using client requested modifications* ".

21. Waste Management

The STAT Analysis Corporation Waste Disposal SOP 1130 identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded in accordance with SOP 1130 Waste Disposal.

22. References

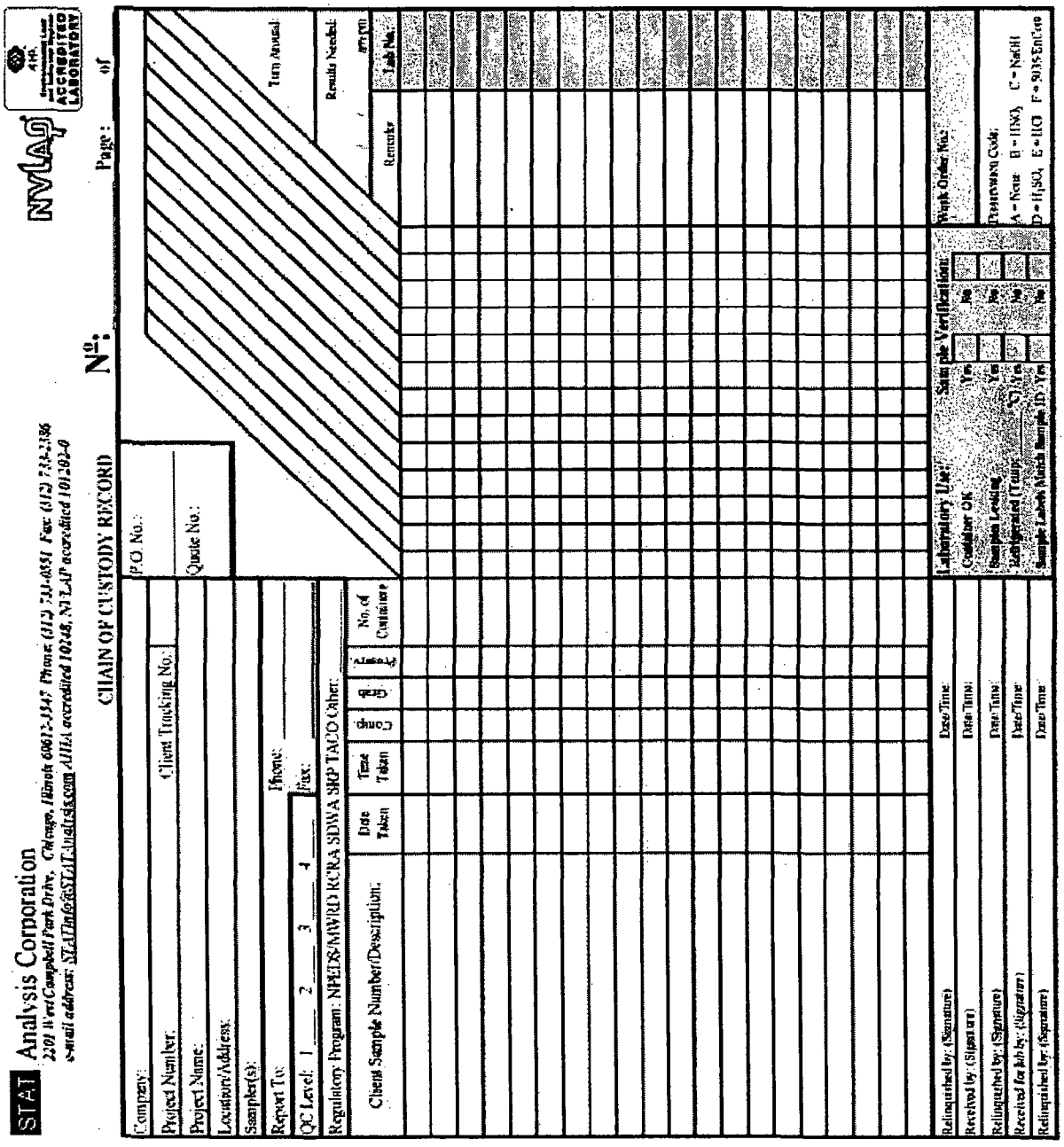
22.1 STAT SOP 1010 Standard and Reagent Preparation
22.2 STAT SOP 220 Customer Service
22.3 STAT SOP 1400 LIMS
22.4 STAT SOP 1130 Waste Disposal
22.5 STAT SOP 003 Chemical Hygiene Plan
22.6 STAT SOP 230 Corrective Actions
22.7 STAT Analysis Corporation Quality Assurance Manual
22.8 Manufacturers' Equipment Instruction Manuals

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

Summary of Attachments:

Attachment 1	Chain-of-Custody Record
Attachment 2	Sample Receipt Checklist Form
Attachment 3	Containers, Preservatives, Holding Times
Attachment 3	Bottle Order Form

ATTACHMENT 1 CHAIN-OF-CUSTODY RECORD



ATTACHMENT 2 SAMPLE RECEIPT CHECKLIST FORM

Water - VOA vials have zero headspace? No VOA vials submitted ☑ Yes ☐ No ☐

Water - pH acceptable upon receipt? Yes ☐ No ☑

Adjusted? _____ Checked by _____

Any No and/or NA (not applicable) response must be detailed in the comments section below.

Client contacted: _____ Date contacted: _____ Person contacted _____

Contacted by: _____ Regarding: _____

Comments: _____

_____

_____

Corrective Action _____

_____

_____

STAT Analysis Corporation

Sample Receipt Checklist

Client Name LFR Date and Time Received: 08/30/02

Work Order Number 0208169 Received by: TMB

Checklist completed by _____ Reviewed by _____

Signature | Date Initials | Date

Matrix: Carrier name: STAT Analysis

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shippping container/cooler?	Yes ☐	No ☐	Not Present ☑
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	

ATTACHMENT 3 CONTAINERS, PRESERVATIVES, HOLDING TIMES

WATER

METALS

Parameter	Container	Volume	Preservative	Holding Time
General, dissolved	Plastic	500 mL	Filtered on site, HNO_3 to pH<2	6 months
General, total	Plastic	500 mL	HNO_3 to pH<2	6 months
Chromium, hexavalent	Plastic	500 mL	Cool 4^0 C	24 hours
Mercury	Plastic	500 mL	HNO_3 to pH<2	28 days

CONVENTIONAL PARAMETERS

Parameter	Container	Volume	Preservative	Holding Time
Acidity	Plastic	500 mL	Cool 4^0 C	14 days
Alkalinity	Plastic	500 mL	Cool 4^0 C	14 days
Ammonia	Plastic	500 mL	H_2SO_4 to pH<2, Cool 4^0C	28 days
BOD	Plastic	500 mL	Cool 4^0 C	48 hours
Bromide	Plastic	500 mL	None	28 days
Chloride	Plastic	500 mL	None	28 days
Chlorine	Plastic	500 mL	Cool 4^0 C	Analyze Immediately
Chromium, Hexavalent	Plastic	500 mL	Cool 4^0 C	24 hours
COD	Plastic	500 mL	H_2SO_4 to pH<2, Cool 4^0C	28 days
Color	Plastic	500 mL	Cool 4^0 C	48 hours
Conductivity	Plastic	500 mL	Cool 4^0 C	28 days

ATTACHMENT 3 CONTAINERS, PRESERVATIVES, HOLDING TIMES (cont)

CONVENTIONAL PARAMETERS

Parameter	Container	Volume	Preservative	Holding Time
Cyanide, Total or Amenable	Plastic	500 mL	NaOH to pH>12, Cool 4° C, (Ascorbic acid if chlorine is present)	14 days
Cyanide, Reactive	Plastic	500 mL	NaOH to pH>12, Cool 4° C	14 days
Fluoride	Plastic	500 mL	None	28 days
Hardness, Total	Plastic	500 mL	HNO_3 to pH<2	6 months
Nitrate/Nitrite	Plastic	500 mL	H_2SO_4 to pH<2, Cool 4°C	28 days
Nitrate	Plastic	500 mL	Cool, 4°C	48 hours
Nitrite	Plastic	500 mL	Cool, 4°C	48 hours
Oil & Grease	Glass	1 liter	H_2SO_4 to pH<2, Cool 4°C	28 days
pH	Plastic	500 mL	None	Analyze Immediately
Phenols	Glass	1 Liter	H_2SO_4 to pH<2, Cool 4°C	28 days
Phosphorus, Ortho	Plastic	500 mL	Cool 4°C, filter on site	48 hours
Phosphorus, Total	Plastic	500 mL	H_2SO_4 to pH<2, Cool 4°C	28 days
Silica	Plastic	500 mL	Cool 4°C	28 days
Solids, Dissolved	Plastic	500 mL	Cool 4°C	7 days
Solids, Suspended	Plastic	500 mL	Cool 4°C	7 days
Solids, Total	Plastic	500 mL	Cool 4°C	7 days
Solids, Settleable	Plastic	500 mL	Cool 4°C	48 hours

ATTACHMENT 3 CONTAINERS, PRESERVATIVES, HOLDING TIMES (cont)

CONVENTIONAL PARAMETERS

Parameter	Container	Volume	Preservative	Holding Time
Solids, Volatile	Plastic	500 mL	Cool 4°C	7 days
Sulfate	Plastic	500 mL	Cool 4°C	28 days
Sulfide	Plastic	500 mL	NaOH to pH>9, Cool 4°C, Zn acetate	7 days
Sulfide, Reactive	Plastic	500 mL	NaOH to pH>9, Cool 4°C	7 days
Sulfite	Plastic	500 mL	None	Analyze Immediately
Surfactants, MBAS	Plastic	500 mL	Cool 4°C	48 hours
Turbidity	Plastic	500 mL	Cool 4°C	48 hours
Total Organic Carbon (TOC)	Plastic	500 mL	H_2SO_4 to pH<2, Cool 4°C	28 days
Total Organic Halogens (TOX)	Glass	1 Liter	H_2SO_4 to pH<2, Cool 4°C	28 days

ORGANICS

Parameter	Container	Volume	Preservative	Holding Time
HPLC Pesticides (Aldicarb / Carbonfuran)	Glass vial	40 mL*	1.2 mL Chloroacetic acid Cool 4°C	28 Days
EDB/DBCP	Glass vial	40 mL*	Cool 4°C	28 Days
Endothall	Glass	1 Liter amber*	Cool 4°C	7 days extraction 1-day analysis
Pesticides and PCBs	Glass	1 Liter amber*	Cool 4°C#	7 days extraction 40 days analysis
Petroleum Hydrocarbons	Glass	1 Liter amber*	H_2SO_4 to pH<2, Cool 4°C	28 days

* Two (2) additional containers needed for QC spiking

Sodium Thiosulfate if chlorine is present

ATTACHMENT 3 CONTAINERS, PRESERVATIVES, HOLDING TIMES (cont)

ORGANICS

Parameter	Container	Volume	Preservative	Holding Time
Phenoxyacid Herbicides	Glass	1 Liter amber*	Cool 4°C#	7 days extraction 40 days analysis
Phthalate Esters	Glass	1 Liter amber*	Cool 4°C	7 days extraction 40 days analysis
Polynuclear Aromatic Hydrocarbons	Glass	1 Liter amber*	Cool 4°C	7 days extraction 40 days analysis
GC/MS Semivolatiles	Glass	1 Liter amber*	Cool 4°C#	7 days extraction 40 days analysis
Total Petroleum Hydrocarbons	Glass	1 Liter amber*	Cool 4°C	7 days extraction 40 days analysis
Volatile Organics	Glass vial	2 x 40 mL*	HCl to pH<2	14 days

* Two (2) additional containers needed for QC spiking

Sodium Thiosulfate if chlorine is present

SOIL

ALL PARAMETERS

Parameter	Container	Volume	Preservative	Holding Time
All except VOA	Glass	2, 4, 8, 32 oz	Cool 4°C	See individual SOP
Volatile Organics	ENCORE (Or equivalent)		Cool 4°C	48 Hours
Volatile Organics	$NaHSO_4$/ Methanol		Cool 4°C	14 Days

STAT Analysis Corporation

ATTACHMENT 4 BOTTLE ORDER FORM

				Drop Off/Pick Up
Client:			Date:	
Contact Name:			Drop Off Address:	
Phone:				
Project Name:			Other Instructions:	
Quantity Needed:	Bottle Type/Preservative:		Quantity Needed:	Bottle Type/Preservative:
	40mL VOA/HCl			500mL Plastic/Unpreserved
	5035 Set			500mL Plastic/HNO_3
	Syringe			500mL Plastic/H_2SO_4
	Green Handle			500mL Plastic/NaOH
	5g Encore			
	25g Encore			
	T-Handle (Metal)			250mL Plastic/Unpreserved
				250mL Plastic/HNO_3
				250mL Plastic/H_2SO_4
	4oz UC/Unpreserved			250mL Plastic/NaOH
	4oz PC/Unpreserved			
	4oz QC/Unpreserved			
	8oz UC/Unpreserved			Cooler
	8oz PC/Unpreserved			Chain of Custody Form
	8oz QC/Unpreserved			Custody Seals
	Quart UC/Unpreserved (Clear)			
				Other:
	2oz UC/Unpreserved			
	2oz UC With Wipe			
	950mL Amber/Unpreserved			
	950mL Amber/H_2SO_4			
			Please Sign and Date Below:	
	950mL Plastic/Unpreserved		Order Taken By:	
	950mL Plastic/HNO_3		Bottle Order Completed By:	
	950mL Plastic/H_2SO_4		Delivery Made:	
	950mL Plastic/NaOH			
			Please Place In Binder When Order Is Complete and Delivered	

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 1000

CONTROL and USE of LABORATORY NOTEBOOKS

Revision 01

Effective Date: December 1, 2004

Author: Laurie Fetterman

Printed Name	Signature/Date
Dennis Jachim **Technical Manager**	_____
Laurie Fetterman **QA Manager**	_____
Thomas M. Bauer **Laboratory Director**	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

Copy Number: _____

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Table of Contents

1. Identification of Test Method

SOP Title: Control and Use of Laboratory Notebooks It may be abbreviated as Lab Notebooks in the laboratory's records. In the current laboratory use of the terms, notebook and logbook have the same meaning.

2. Applicable Matrix or Matrices

Not Applicable to the SOP

3. Detection Limits

Not Applicable to the SOP

4. Scope and Application

This SOP details the procedures used to create and control the notebooks used throughout the testing laboratory and related departments of STAT Analysis Corporation. It also details the procedures that each employee must follow to record original observations in order to provide uniform entry of information into the laboratory notebooks. Each notebook will have the same attributes and format to ensure that all pertinent information for historical reconstruction of the laboratory's testing related activities is complete and serves as a "stand alone" record. Testing related activities encompass the laboratory's production of data related to individual test methodology, the ancillary support services, and the sustaining records of sample condition.

5. Summary of Test Method

Laboratory notebooks may either be purchased from an outside vendor or created in the laboratory using in-house word processing capabilities. The notebooks are issued as controlled numbered recording devices. Data is entered on each page as appropriate. Once filled, or replaced by an updated edition, the notebook is taken out of service and archived. A new controlled notebook with a new number is issued. The archived notebook is placed in storage. The QA manager is responsible for the creation, distribution, control, and archiving of laboratory notebooks.

The procedures to be followed for laboratory notebooks are outlined in Section 14 of this SOP. They include the following:

1. Creation
2. Contents
3. Assigning IDs and Control Numbers
4. Distribution
5. Usage
6. Review
7. Requests (for New Notebooks)

8. Collection
9. Archiving

Real-time notebook review is performed at the department level. If the department level review reveals a deficiency in any area of notebook content or usage, the corporation's corrective action process, SOP 230 Corrective Action, is employed.

The QA Manager, as part of the Internal QA Audit process, SOP 1220 Internal Quality Assurance Audit, also reviews the laboratory notebooks to ensure test method SOP requirements and employee compliance. This is done as a historical review usually performed post data release. When the QA manager notes deficiencies during his/her internal audit of laboratory records, the corporation's corrective action process is employed to remedy these situations. The appropriate Department Manager is tasked to take immediate corrective action and to implement the necessary changes in an expedient manner. Additional audits and follow up audits are performed as necessary to verify that corrective actions have been implemented and are successful in correcting the respective deficiencies.

Additional documentation is through the use of the Corrective Action Report (SOP 230 Corrective Action).

6. Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

Not Applicable to the SOP

8. Safety

Not Applicable to the SOP

9. Equipment and Supplies

Not Applicable to the SOP

10. Reagents and Standards

Not Applicable to the SOP

11. Sample Collection, Preservation, Shipment and Storage

Not Applicable to the SOP

12. Quality Control

Not Applicable to the SOP

13. Calibration and Standardization

Not Applicable to the SOP

14. Procedure

14.1 Creation of Laboratory Notebooks

Laboratory notebooks may either be purchased from an outside vendor or created in the laboratory using in-house word processing capabilities.

14.1.1 Purchased notebooks are customized for the intended purpose (drawing columns, adding column header information, etc.) and must have each page paginated.

14.1.2 For in-house notebooks, a customized template sheet is created for a particular test methodology or support function. The template is stored as a limited access electronic file. It is used to create new copies of the notebook until a revised template is created. The template is used to produce multiple copies. The copies are gathered and paginated. The pages are bound using spine binders. A plastic front and back cover is attached to protect the notebook.

14.1.3 Each type of notebook must have a cover page. The cover page includes the following: Notebook number, title, department or area location, SOP number if applicable, effective dates, and a signature receipt line.

14.1.4 The QA Manager reviews the bound notebook to ensure that all appropriate pages are included, the notebook is complete, and ready for service.

14.1.5 Customized forms: These forms may be purchased or created in-house. Forms are used to record information for a particular set of samples (Chain-Of-Custody) or for a particular laboratory activity (Sample Cooler Receipt Form, intra laboratory communications forms that contain information relevant to the testing activities, etc.). These forms usually are kept in a job folder and are eventually filed with the final report. The QA Manager reviews and controls the current versions in use. Current forms are assigned a title and a control number (or effective date). Laboratory employees may create their own copies of these forms on an as needed basis. The QA Manager must first approve any revisions prior to use. A new controlled revised form is then issued. The QA Manager archives the outdated form. The steps for control and use of these customized forms are performed in a similar manner as outlined in the following sections of this SOP.

14.1.6 Electronic records: Software used to produce records for data reduction, validation, storage and reporting are addressed in SOP 1500 Computer Network.

14.2 Contents of Laboratory Notebooks

Contents of each laboratory notebook will be appropriate to the particular test method or laboratory operation. All required information must be recorded in the notebook. The information recorded in

the notebook must be sufficient and complete to allow the historical reconstruction of laboratory's testing related activities. The following information is not all-inclusive. Individual SOPs contain the required information as pertinent to the procedure.

14.2.1. Record of sample condition: sample preservation, appropriate container, holding time compliance, Sample ID, receipt, acceptance or rejection, log-in, sample storage and tracking shipping receipts.

14.2.2. Essential information and raw data includes: Sample ID, date/time of analysis, instrument ID and operating conditions, method ID, manual integrations and calculations, analyst's signature or initials; sample prep information to include procedure, sample weights/volumes, appropriate units, ID codes, instrument printouts and meter readings, calculations, reagents used; sample analysis; standard and reagent origin, receipt, preparation, and use; calibration criteria, frequency and acceptance criteria; data and statistical calculations, review, confirmation, assessment, and reporting conventions; QC protocols and assessment; method performance criteria including expected QC requirements

14.2.3. Any additional comments or observations that would aid in the future reconstruction of the data.

14.2.4. Maintenance Notebooks: For instrument maintenance notebooks, the format should consist of the following categories: "Problem" (define clearly), "Action Taken" (state all steps taken, note if outside service vendor was used to correct the problem, add reference number or attain signature of outside vendor), and "Verification" (initials and date, ensure that the problem has been corrected and that the instrument was successfully recalibrated after the maintenance procedure).

14.3 Assigning IDs and Control Numbers to Laboratory Notebooks

The following is a responsibility of the QA Manager.

14.3.1. Prior to distribution to the laboratory department, each notebook is labeled with a control number, a title, department or area location, and an SOP number if applicable assigned by the QA Manager. A field is provided that requires the effective dates of usage (defined as the first and last dates that data and/or observations were recorded into the notebook). A signature line is provided to record the employee receiving the notebook.

14.3.2. The QA Manager maintains and controls a master list of all notebooks distributed to the laboratory departments. This list includes the notebook control number, title, department or area location, SOP number if applicable, effective dates, recipient name, status (active or archived), and collection date (archive date).

14.4 Distribution of Laboratory Notebooks

The following is a responsibility of the QA Manager.

14.4.1 The QA Manager delivers the notebook to the appropriate department.

14.4.2 The person receiving the notebook signs his name to indicate receipt of the notebook.

14.4.3 The QA Manager updates the master list to record the new notebook distribution.

14.5 Usage of Laboratory Notebooks

Employees must comply with the following measures to ensure complete and comprehensible records.

14.5.1. Clear identification of personnel involved with sample handling, preparation, calibration, testing (signatures or initials) must be evident.

14.5.2. Dates and Times, as appropriate, are clearly recorded.

14.5.3. All handwritten entries must be legibly recorded in permanent ink.

14.5.4. No obliterations, cross-outs, whiteouts, or deletions are made to the records.

14.5.5. Corrections to records are made by drawing one line through the data, recording the amended data, applying initials and date.

14.5.6. All calculations, including calculations for dilutions are recorded.

14.5.7. Page remnants and pages not used are crossed off with a single line applied diagonally, initialed and dated.

14.5.8. Corrective actions (CAR number) are noted; Quality Exception Report (QER number) (if applicable) is noted.

14.5.9. Appropriate data flags are used, if applicable.

14.5.10. Notebooks are not exposed to water or laboratory chemicals that could jeopardize the condition of the notebook. When not in use, notebooks are stored in an appropriate, secure area of the department.

14.5.11. Notebooks should only be handled by the person using the notebook to record data or a person reviewing the data entries.

14.5.12. Post-it notes ("sticky notes"), scraps of paper, or unauthorized forms are not to be used to record any pertinent information that becomes part of the permanent record.

14.5.13. It is not permitted to remove notebooks from the department unless prior permission is obtained from the Department Manager. They may be transported from one department to another for information or photocopying purposes. It is not permitted to remove active notebooks from the building unless special permission is granted from top management.

14.6 Review of Laboratory Notebooks

The review process ensures that the recorded data has been assessed for quality, accuracy, and completeness. The following is a responsibility of the Department Manager and the QA Manager.

14.6.1 It is the responsibility of the Department Manager, or the person assigned to perform the review, to ensure that their employees are in compliance with the requirements as detailed in Section 14.5.

14.6.2 SOP 1250 Data Review contains the details for data review for the analytical test methods, the sample preparation methods, and laboratory support functions.

14.6.3 The data on each page of the notebook must be reviewed and signed-off by a secondary reviewer. Note: if the data in the notebook undergoes secondary review as detailed in SOP 1250 Data Review and the review is documented on a checklist, secondary sign-off is not required for each page.

14.6.3.1 The following notebooks are not part of the secondary review process in that there is no secondary review checklist associated with them. Secondary notebook review and sign-off of each page is required for the following notebooks:

Instrument Maintenance Notebooks
Wet Chemistry Notebooks
Standards and Reagents Notebooks
Balance, Thermometer, and Autopippettes Calibration Notebooks

14.6.4 The QA Manager, as part of the Internal Audit process SOP 1220 Internal Quality Assurance Audit, also reviews the laboratory notebooks to ensure employee compliance.

14.7 Requests for New Laboratory Notebooks

The following is a responsibility of the Department Manager.

14.7.1 Prior to the completion of all pages of the notebook, the Department Manager (or other employee responsible for using the notebook as assigned by the Department manager) submits a request to the QA Manager for a new notebook. The request may be written (as an email or memo) or verbal. The written request is preferable.

14.7.2 The QA Manager fills the request by following the steps in this SOP. The new notebook is created and distributed in a timely manner in order to provide the new notebook prior to the completion of the current notebook. This will prevent a stoppage of production or the possible use of a non-controlled means of recording pertinent information.

14.8 Collection of Laboratory Notebooks

The following is a responsibility of the QA Manager.

14.8.1 Once filled, or replaced by an updated edition, the notebook is taken out of service.

14.8.2 The completed notebook may reside in the respective department or laboratory area for a period of time not to exceed six months. This is done to accommodate the department employees who may use the completed notebook as a reference for recently completed testing.

14.8.3 The QA Manager removes the notebook from the department. After it is collected, the QA Manager archives the notebook.

14.9 Archiving of Laboratory Notebooks

The following is a responsibility of the QA Manager.

14.9.1. After collection, the notebook is archived and placed in secure storage. See SOP 240 Archiving.

14.9.2. The QA Manager updates the master list of all notebooks. The effective dates are recorded, the status is updated to archived, and the archive date is recorded.

14.9.3. The notebooks remain in archived storage for a minimum of five (5) years after the last entry in the notebook.

14.9.4. Customized forms: The original forms are kept in an archive folder. They remain in archived storage for a minimum of five (5) years.

15. Data Reduction, Calculations and Loading

Not Applicable to the SOP

16. Method Performance

Not Applicable to the SOP

17. Pollution Prevention

Not Applicable to the SOP

18. Data Assessment and Criteria for Quality Control Measures

Not Applicable to the SOP

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the Notebook Review findings determine that the laboratory performance for a particular parameter is judged to be out of control, the problem must be immediately identified and corrected. The

analytical results produced for that parameter are suspect. The time frame for the out of control situation will be determined. This will identify what client samples were analyzed during the time frame. Immediate corrective action includes written notification to clients that the data produced for the parameter may be affected and, if possible, to the degree that the data was affected.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data are revealed through the Notebook Review process, the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discuss options with the client. Reanalysis or reporting the data with qualification are alternatives. This may include the re-issue of amended reports with qualified data indicating that the previously reported results did not meeting the laboratory defined criteria. Out-of-control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting. For amended reports, the reason for the report amendment is clearly identified and explained.

Final data results must be qualified in the client report for results not meeting the laboratory-defined criteria.

The process for handling unacceptable and out-of-control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and recorded by the Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21. Waste Management

Not Applicable to the SOP

22. References

22.1 STAT Analysis Corporation Quality Assurance Manual
22.2 SOP 230 Corrective Actions
22.3 SOP 240 Archiving
22.4 SOP 1220 Internal Quality Assurance Audit
22.5 SOP 1250 Data Review
22.6 SOP 1500 Computer Network
22.7 STAT SOPs [all test methods]
22.8 STAT SOPs [all support equipment, record keeping, MDL's, etc]
22.9 NELAC, Chapter 5, current revision at time of approval.

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

Not Applicable to the SOP

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 1010

ANALYTICAL STANDARDS and REAGENTS RECEIPT and PREPARATION

Revision 01
Effective Date: December 8, 2004

Author: Laurie Fetterman

Printed Name	Signature/Date
Dennis Jachim **Technical Manager**	_____
Laurie Fetterman **QA Manager**	_____
Thomas M. Bauer **Laboratory Director**	_____

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Table of Contents

1. Identification of Test Method

SOP Title: Analytical Standards and Reagents Receipt and Preparation is abbreviated as Stds Prep and Reagent Prep in the laboratory's records.

2. Applicable Matrix or Matrices

Not Applicable to the SOP

3. Detection Limits

Not Applicable to the SOP

4. Scope and Application

This SOP details the procedures used to receive, identify, record, prepare, and control the analytical standards and reagents used throughout the testing laboratory and related departments of STAT Analysis Corporation. It also details the procedures that each employee must follow to record original observations in order to provide uniform entry of information into the laboratory standards and reagents notebooks. The standards and reagents notebooks for each type of test method will have the same attributes to ensure that all pertinent information for historical reconstruction of the laboratory's testing related activities is complete and easily understood. The notebook format is customized according to its function. Testing related activities encompass the laboratory's production of data related to individual test methodology, the ancillary support services, and the sustaining records of sample condition.

5. Summary of Test Method

Analytical standards and reagents are purchased from an outside vendor and received in the laboratory. The items are checked upon receipt to ensure the correct item, as purchased, is received. Items are inspected to ensure that they are free from damage or defect. Customized laboratory notebooks are used to record information about the materials as received and the subsequent use of these materials to create other standard solutions and test method reagents used throughout the laboratory. A unique ID number is assigned to the standard or reagent upon receipt. A unique ID number is also assigned and to any subsequent preparation of solutions. The ID number is entered into the laboratory's records each time the material is used. Information is entered on each page of the standards or reagents notebook as appropriate. The information must be sufficient to enable the tracking of the standard or reagent from its source to any use within the laboratory's testing related activities. Expired and outdated materials are collected, removed from service, and disposed. The Department Managers and the QA Manager are responsible for employee compliance to this SOP.

Additional information on laboratory notebooks may be found in SOP 1000 Control and Use of Laboratory Notebooks.

The procedures to be followed for analytical standards and reagents receipt and preparation are outlined in Section 14 of this SOP. They include the following:

1. Receipt and Inspection
2. Assigning ID Numbers

3. Notebooks
4. Preparation and Labeling
5. Validation
6. Usage
7. Review
8. Requests (for new materials)
9. Collection, Removal, and Disposal

Real-time notebook review is performed, using the data review process SOP 1250, at the department level. If the department level review reveals a deficiency in any area of notebook content or usage, the corporation's Corrective Action process, SOP 230, is employed.

The QA Manager, as part of the Internal QA Audit process, SOP 1220, also reviews the laboratory notebooks to ensure test method SOP requirements and employee compliance to this SOP. This is done as a historical review usually performed post data release. When deficiencies are noted by the QA Manager during his internal audit of laboratory records, the corporation's corrective action process is employed to remedy these situations. The appropriate Department Manager is tasked to take immediate corrective action and to implement the necessary changes in an expedient manner. Additional audits and follow up audits are performed as necessary to verify that corrective actions have been implemented and are successful in correcting the respective deficiencies.

Additional documentation is through the use of the Corrective Action Report (SOP 230 Corrective Action).

6. Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

Not Applicable to the SOP

8. Safety

Safety glasses, gloves, lab coats and closed toe shoes, are a minimum.

Special care must be taken when handling large bottles of solvents and concentrated acids and bases. Carry only one large bottle at a time using both hands to carry and steady the weight.

A hand truck should be used to transport cases of reagents and gas cylinders.

Use proper lifting techniques when lifting cases of reagents and handling gas cylinders; they are heavy.

Other safety precautions must be conducted in accordance with the SAP 003 Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of material safety data sheets (MSDS) is available in each room for personnel involved in an analysis using chemicals.

9. Equipment and Supplies

Not Applicable to the SOP. See individual test method SOP's for specific equipment and supplies.

10. Reagents and Standards

Not Applicable to the SOP

11. Sample Collection, Preservation, Shipment and Storage

Not Applicable to the SOP

12. Quality Control

Not Applicable to the SOP

13. Calibration and Standardization

Not Applicable to the SOP

14. Procedure

14.1 Receipt and Inspection of Analytical Standards and Reagents

Analytical Standards and Reagents are purchased from qualified outside vendors. The purchased material may be pure chemicals, solutions and mixtures of chemicals, or cylinders of compressed gases.

14.1.1 The procedure for receipt and inspection is detailed in SOP 1330 Purchasing Section 14.4.

14.1.2 Once delivered to the appropriate department, the material is inspected to ensure that it meets the minimum quality standards for which it will be used. Any certificates, pertaining to material traceability and condition, that accompany the material are filed in the department in an appropriate folder or notebook. Certificates are labeled with receipt date and the associated material unique ID number (see section 14.2). If no certificate was received for a calibration standard, the vendor must be contacted to obtain the information.

14.1.3 Analytical standards and reagents must be stored according to the manufacturer's recommended storage condition. If the storage condition is not stated on the container or the associated certificate, refer to the applicable SOP for guidance on proper storage condition requirements.

14.2 Assigning ID Numbers to Analytical Standards and Reagents

The following is the responsibility of the Department Manager.

14.2.1. Prior to using the purchased material, a Unique ID Number must be assigned. This number is used to track the usage of the material throughout the laboratory. It is also used to identify the source material

used to prepare subsequent solutions. Each container of the received material that is labeled with the same lot number, may be assigned the same Unique ID Number. Multiple containers may be assigned a designation as "1" of "3", etc.

14.2.2. Prior to using the laboratory prepared solutions, a Unique ID Number must be assigned. This number is used to track the usage of the material throughout the laboratory. It is also used to identify the source material used to prepare any additional subsequent solutions (that are also assigned Unique ID Numbers). A prepared solution may be divided into separate storage containers. Each container may be labeled with the same Unique ID Number. Multiple containers may be assigned a designation as "1" of "3", etc.

14.3 Notebooks for Analytical Standards and Reagents

Contents of each laboratory notebook will be appropriate to the particular test method or laboratory operation. All required information must be recorded in the notebook. The information recorded in the notebook must be sufficient and complete to allow the historical reconstruction of laboratory's testing related activities. The following information is not all-inclusive. Individual SOPs may require additional information as pertinent to the procedure.

14.3.1. For Analytical Standards and Reagents as received, record the following:

14.3.1.1. Unique ID Number,
14.3.1.2. Name of Material,
14.3.1.3. Manufacturer,
14.3.1.4. Lot Number,
14.3.1.5. Purity of Material (if less than 96% pure) or Concentration of Material in Solution,
14.3.1.6. Expiration Date,
14.3.1.7. Storage Condition/Location,
14.3.1.8. Number of Containers,
14.3.1.9. Date Received,
14.3.1.10. Initials (of the employee entering the initial information into the notebook),
14.3.1.11. Date Opened,
14.3.1.12. Initials (of the employee opening the container),
14.3.1.13. Date Removed from Service (due to expiration of the material or the material was entirely consumed: note as such in the notebook),
14.3.1.14. Initials (of the employee removing the container from the department).
14.3.1.15. Date of Validation (if the material is used as received in the test method).
14.3.1.16. Initials (to indicate acceptability of the material).

14.3.2. For Calibration Standards, QC check solutions, and Test Method Reagents that must be prepared in the laboratory, record the following:

14.3.2.1. Unique ID Number,
14.3.2.2. Name of Prepared Standard or Solution,
14.3.2.3. Solution prep information to include the procedure and storage condition/location (or reference to the procedure and storage condition/location in the applicable SOP),
14.3.2.4. Unique ID Number(s) of the source material(s),
14.3.2.5. Sample Volumes/Weights of the source material(s),
14.3.2.6. Final Volumes/Weights of the prepared solution/mixture,

14.3.2.7. Concentration,
14.3.2.8. Appropriate Units,
14.3.2.9. Expiration Date,
14.3.2.10. Number of storage containers (if applicable),
14.3.2.11. Date Prepared,
14.3.2.12. Initials of preparer,
14.3.2.13. Date of Validation,
14.3.2.14. Initials (to indicate acceptability of the solution),
14.3.2.15. Date Removed from Service (due to expiration of the material, the material was entirely consumed, or the material was replaced with a subsequent preparation: note as such in the notebook),
14.3.2.16. Initials (of the employee removing the container from the department),
14.3.2.17. Balance ID, as applicable
14.3.2.18. Pipette ID, as applicable

14.3.3. For preparations of calibration standards and QC check solutions, the concentration of the original source material is required. This may be referenced in the SOP if the concentration of the source material remains unchanged.

14.3.4. Preparations of calibration standards and QC check solutions using source materials of < 96% purity must be corrected using the stated purity of the material.

14.3.5. Calibrated instruments, such as balances and autopipettes, used in the preparation of solutions must be identified using the instrument ID or serial number.

14.3.6. Expiration Dates: refer to the manufacturer's stated expiration date. If not stated, review the specific test method SOP for guidance. In general, dry pure inorganic chemicals and concentrated acids and bases have a default expiration date of five (5) years from date of opening. Organic solvents and aqueous solutions have a default expiration date of one (1) year from date of opening. Expiration dates may be shorter than this criteria depending upon the material's stability. The expiration date may be extended if a stability study indicates the material is still of sufficient quality to be used in the testing process. The stability study must be recorded and approved by the QA Manager or the Technical Director. The material must be remarked to indicate the new expiration date.

14.3.6.1. The expiration date of a laboratory prepared analytical standard or reagent (Section 14.3.2.9) cannot exceed the expiration date of any stock analytical standard or reagent used in its preparation.

14.3.7. The completed standards or reagent notebooks may reside in the respective department or laboratory area for a period of time This is done to accommodate the department employees who may use the completed notebook as a reference for recently completed testing. After collection, the notebook is archived and placed in secure storage. See SOP 240 Archiving.

14.4 Preparation and Labeling of Calibration Standards, QC Solutions and Test Method Reagents

The following is the responsibility of the employee performing the procedure.

14.4.1 Some materials are used as received in the laboratory. If the material remains in the original container, the following minimum information must be added to the container label:

14.4.1.1 Unique ID Number
14.4.1.2 Expiration Date (if not already stated)
14.4.1.3 Date Received
14.4.1.4 Date Opened
14.4.1.5 Initials (of the employee opening the container)
14.4.1.6 Container number (optional)

14.4.2 Instructions for solutions preparation are in each test method SOP. Instructions may also be listed in the front of the preparation notebook or on each page of the preparation notebook as appropriate.

14.4.3 The person performing the procedure must follow the procedure exactly as written.

14.4.4 The prepared solution or reagent container must be labeled with the following minimum information:

14.4.4.1. Unique ID Number
14.4.4.2. Name
14.4.4.3. Concentration
14.4.4.4. Appropriate Units
14.4.4.5. Expiration Date
14.4.4.6. Date Prepared
14.4.4.7. Initials of preparer
14.4.4.8. Number of storage containers (optional)

14.4.5 The prepared solution or reagent may be transferred to a smaller container for ease of handling or for safety reasons. If this is done and the material in the smaller container is consumed within the shift period or up to 24 hours, only the unique ID number and name of the material are required on the small container.

14.4.6 The person performing the procedure must record all required information and initial the notebook. The person (or a designee)performing or reviewing the validation procedure must initial the notebook.

14.4.7 Modifications to the procedure: any modifications or changes must be reviewed and approved by the Department Manager, and the QA Manager or the Technical Director. Changes in volumes of solutions prepared (increased or decreased) may be approved by the Department Manager if the proportions of the materials remain the same and the accuracy of source material measurement is not affected.

14.5 Validation of Analytical Standards and Reagents

All purchased Analytical Standards, Reagents, and Prepared Solutions must be validated prior to use in the laboratory. This is done to prevent the introduction of a material into the test method or sample handling procedure that may affect the quality of the sample test result.

14.5.1 The validation procedure is test method dependent.

14.5.2 Calibration standards must be verified using a second source confirmation procedure.

14.5.3 For those test methods that use titrants to analyze sample components, the titrant concentration must be independently verified according to reference method procedures or other laboratory protocol.

14.5.4 QC solutions must be verified using an independent analysis.

14.5.5 Reagents must be verified using the appropriate test method. The usual procedure is to analyze a Method Blank and a known QC solution to verify contamination level and method performance.

14.6 Usage of Analytical Standards and Reagents

Employees must comply with the following measures to ensure complete and comprehensible records.

14.6.1 Clear identification of the Unique ID Number must be evident with all sample handling, preparation, calibration, and testing records.

14.6.2 The material is used only for its intended purpose as detailed in the applicable SOP.

14.6.3 Employees may not reuse any ID Number for purchases of different materials or for preparations of solution and reagents. New materials, marked with the same manufacturer's lot number, may be marked with the same Unique ID. A new entry must be made in the logbook to record the information listed in Section 14.3.1. The material, if used as received for a particular application, may be immediately placed in service, bypassing the validation procedure. However, any prepared solutions, using this new material as the source material, must still be validated.

14.6.4 The material may be used until the expiration date or until it is replaced by a different material or solution.

14.6.5 The expiration date may be extended if a stability study indicates the material is still of sufficient quality to be used in the testing process. The stability study must be recorded and approved by the QA Manager or the Technical Director. The material must be remarked to indicate the new expiration date.

14.7 Review of Analytical Standards and Reagents

The review process ensures that the recorded data has been assessed for quality, accuracy, and completeness. The following is the responsibility of the Department Manager and the QA Manager.

14.7.1 It is the responsibility of the Department Manager, or the person assigned to perform the review, to ensure that their employees are in compliance with the requirements as detailed in this SOP.

14.7.2 SOP 1250 Data Review contains the details for data review for the analytical test methods, the sample preparation methods, and laboratory support functions. The traceability of standards and reagents is part of the review.

14.7.3 The data on each page of the notebook must be reviewed and signed-off by a secondary reviewer. Note: if the data in the notebook undergoes secondary review as detailed in SOP 1250 Data Review and the review is documented on a checklist, secondary sign-off is not required for each page.

14.7.4 The QA Manager, as part of the Internal QA Audit process SOP 1220, also reviews the laboratory notebooks to ensure employee compliance.

14.8 Requests for New Analytical Standards and Reagents

The following is the responsibility of the Department Manager.

14.8.1 Prior to the complete usage of the standard or reagent, the Department Manager (or other employee responsible for using the material as assigned by the Department Manager) submits a request to the purchasing agent for the new material. The request may be written (as an email or memo) or verbal. A verbal request is sufficient for standing and repeat orders of the same material from the same supplier. See SOP 1330 Purchasing for additional information.

14.8.2 The Department Manager must provide adequate time to execute the purchase, the receipt, and the validation of the new material prior to the exhaustion of the current material in use. This will prevent a stoppage of production or the possible use of non-authorized material.

14.9 Collection, Removal, and Disposal of Analytical Standards and Reagents

The following is the responsibility of the Department Manager, QA Manager or the person in charge of sample disposal, to remove the standard or reagent from service.

14.9.1 Collection and removal occur due to the following: expiration of the material, the material was entirely consumed (empty container is removed), or the material was replaced with a subsequent preparation. The Date Removed from Service, and Initials (of the employee removing the container from the department) are recorded in the notebook.

14.9.2 The material is physically removed from the department and placed in the appropriate location per SOP 1130 Waste Disposal. Only authorized employees may handle the material at this point.

14.9.3 If not disposed, expired standards are placed in a secure area with limited access. These expired standards can be either re-certified or used for qualitative purposes.

15. Data Reduction, Calculations and Loading

Calculations involve basic weight to volume or volume to volume ratios.

16. Method Performance

Not Applicable to the SOP

17. Pollution Prevention

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the lab waste disposal program, SOP 1130 Waste Disposal.

18. Data Assessment and Criteria for Quality Control Measures

Not Applicable to the SOP

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the Analytical Standards and Reagents Notebooks Review findings determine that the laboratory performance for a particular parameter is judged to be out-of-control, the problem must be immediately identified and corrected. The analytical results produced for that parameter are suspect and are only reported for regulatory compliance purposes with the appropriate corrective action form. The time frame for the out-of-control situation will be determined. This will identify what client samples were analyzed during the time frame. Immediate corrective action includes written notification to clients that the data produced for the parameter may be affected and, if possible, to the degree that the data was affected. Immediate corrective action may include reanalyzing all affected samples by using any retained sample before the expiration of the holding time. Final data results must be qualified in the client report for reported results not meeting the laboratory-defined criteria.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data are revealed through the Analytical Standards and Reagents Notebooks Review process, the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discuss options with the client. Reanalysis or reporting the data with qualification are alternatives. This may include the re-issue of amended reports with qualified data indicating that the previously reported results did not meeting the laboratory defined criteria. Out-of-control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting. For amended reports, the reason for the report amendment is clearly identified and explained.

Final data results must be qualified in the client report for results not meeting the laboratory-defined criteria.

The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and recorded by the Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21. Waste Management

See Reference SOP 1130 Waste Disposal

22. References

22.1 STAT Analysis Corporation Quality Assurance Manual
22.2 SAP 003 Chemical Hygiene Plan
22.3 SOP 230 Corrective Actions
22.4 SOP 1000 Control and Use of Laboratory Notebooks
22.5 SOP 1130 Waste Disposal
22.6 SOP 1220 Internal Quality Assurance Audit
22.7 SOP 1230 Training
22.8 SOP 1250 Data Review
22.9 SOP 1330 Purchasing
22.10 NELAC Chapter 5 Quality Systems, current version at time of approval

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

ATTACHMENT 1: Stock Standards and Reagents (Original Materials)
ATTACHMENT 2: Working Standards and Reagents (Laboratory Prepared Solutions) –Inorganic Department
ATTACHMENT 3: Working Standards and Reagents (Laboratory Prepared Solutions) –Organic Department

ATTACHMENT 1: STOCK STANDARDS AND REAGENTS: *Department*

(Original Material)

Logbook #: XX-XXXX

Unique ID Number					
Name of Material					
Manufacturer					
Lot Number					
Purity (if < 96%)					
Concentration					
Expiration Date					
Storage Condition Location					
# of Containers					
Date Received					
Initials (enter info)					
Date Opened					
Initials (open)					
Date Removed (EXP or CONS)					
Initials (removal)					
Date of Validation					
Initials (accept)					

Unique ID Number					
Name of Material					
Manufacturer					
Lot Number					
Purity (if < 96%)					
Concentration					
Expiration Date					
Storage Condition Location					
# of Containers					
Date Received					
Initials (enter info)					
Date Opened					
Initials (open)					
Date Removed (EXP or CONS)					
Initials (removal)					
Date of Validation					
Initials (accept)					

CODES:_____

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ATTACHMENT 2: WORKING STANDARDS AND REAGENTS: *Inorganic Department*

(Laboratory Prepared Solutions) *Logbook #: XX-XXXX*

Unique ID Number					
Name of Solution					
Prep Reference					
1. Source ID # Vol/Wt Source					
2. Source ID # Vol/Wt Source					
3. Source ID # Vol/Wt Source					
4. Source ID # Vol/Wt Source					
5. Source ID # Vol/Wt Source					
6. Source ID # Vol/Wt Source					
7. Source ID # Vol/Wt Source					
8. Source ID # Vol/Wt Source					
Final Vol/Wt					
Conc & Units					
Expiration Date					
Storage Condition Location					
# of Containers					
Date Prepared					
Initials (preparer)					
Date of Validation					
Initials (accept)					
Date Removed (EXP or CONS)					
Initials (removal)					
Balance ID					
Pipette ID					

CODES:_____

Prep Procedure:_____

_____

_____

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ATTACHMENT 3: WORKING STANDARDS AND REAGENTS: *Organic Department*

Logbook #: XX-XXXX

WORKING STANDARDS AND REAGENTS: Organics

(Laboratory Prepared Solutions)

Unique ID Number					
Name of Solution					
Prep Reference					
1. Source ID Vol/ Wt.of Source					
2. Source ID Vol/ Wt.of Source					
3. Source ID Vol/ Wt.of Source					
4. Source ID Vol/ Wt.of Source					
5. Source ID Vol/ Wt.of Source					
6. Source ID Vol/ Wt.of Source					
7. Source ID Vol/ Wt.of Source					
8. Source ID Vol/ Wt.of Source					
9. Source ID Vol/ Wt.of Source					
Final Vol/ Wt.					
Conc. & Units					
Date Prepared					
Initials (preparer)					
Expiration Date					
Storage Condition/location					
# of Containers					
Date of Validation					
Initials (accept)					
Date Removed (EXP or CONS)					
Initials (removal)					

CODES: _____

Prep. Procedures: _____

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Secondary Reviewer/Date: _____

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 1250

DATA REVIEW

Revision 01
Effective Date: November 17, 2004

Author: Laurie Fetterman

Printed Name	Signature/Date
Dennis Jachim **Technical Manager**	_____
Laurie Fetterman **QA Manager**	_____
Thomas M. Bauer **Laboratory Director**	_____

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Table of Contents

1. Identification of Test Method

SOP Title: Data Review

2. Applicable Matrix or Matrices

Not Applicable to the SOP

3. Detection Limits

Not Applicable to the SOP

4. Scope and Application

This SOP details the procedures used to perform Data Review. The review, conducted using a three level review process, encompasses the laboratory's production of data related to individual test method batches, to ancillary support services, and to sustaining records of sample condition. The review process ensures that the reported data has been assessed for quality, accuracy, and completeness.

The Data Review process is used to substantiate that the data production aspect of the quality system is appropriate and effective to the current level of laboratory activity.

5. Summary of Test Method

The Data Review process requires that three independent levels of review be performed prior to release of the data. The first level is performed by the analyst or technician who completes the initial analysis or sample preparation process (data batch). The second level is performed by either a Supervisor or Department Manager in the respective laboratory department, or by a peer analyst trained in the procedure that was used to produce the data batch. The third level of review is performed by a Project Manager or by the person authorizing the release of the data in the final report. This third level of review is considered an overall check of the data involving an overview to check that all sample analyses were performed as requested. It is also a check to determine if the sample was received in a proper condition and that it was analyzed within holding time. If applicable, there may be opportunities to compare different types of tests, on the same sample, to determine if results confirm one another, such as PNA analysis compared with SVOC analysis or the Cr^{+6} analysis compared to the total Cr analysis.

The criteria to be checked are outlined in Section 14 of this SOP. They may include the following:

1. Initial Calibration (ICAL), including Mass Spec tuning or instrument (GC) performance
2. Continuing Calibration Verification (CCV), including Mass Spec tuning or GC performance
3. Method Blanks (MB)
4. Laboratory Control Samples (LCS/LCSD)
5. Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
6. Laboratory Duplicates
7. Surrogates
8. Internal Standards
9. Other Method specific QC samples (such as post digestion spikes)

10. Sample results
11. Sample dilutions
12. Manual calculations
13. Manual integrations
14. Reporting Limits (RL)
15. Holding time criteria
16. Sample preservation criteria
17. Sample preparation criteria, including support equipment calibration
18. Sample storage criteria
19. Completeness of records
20. Corrective actions for QC failures
21. Any additional comments or observations that would aid in the future reconstruction of the data

The review is recorded by the secondary reviewer signing and dating a page of a notebook or by the use of checklists customized for each area of the laboratory and its particular test methodology. Each person performing the first level review and the second level review is required to sign his name or initials and record the date of the review on the checklist. The third level of review is not part of the data review checklist. The signature of the Project Manager or Laboratory Director on the final report affirms his review of the data. All data must be reviewed in this manner prior to release.

If the review reveals a deficiency in any area of data production, the secondary reviewer will return the notebook or the data packet to the analyst with the appropriate comments to take corrective action such as re-analysis, reintegration, or additional review.

The QA Manager, as part of the Internal QA Audit process, reviews 5% of the data produced in the laboratory. One of the tools used in this audit process is the data review checklist. When deficiencies are noted by the QA manager during his internal audit of data batches, the corporation's corrective action process is employed to remedy these situations. The appropriate Department Manager is asked to take immediate corrective action and to implement the necessary changes in an expedient manner. Additional audits and follow up audits are performed as necessary to verify that corrective actions have been implemented and are successful in correcting the respective deficiencies.

Additional documentation is through the use of the Corrective Action Report (STAT SOP 230 Corrective Action).

6. Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

Not Applicable to the SOP

8. Safety

Not Applicable to the SOP

9. Equipment and Supplies

Not Applicable to the SOP

10. Reagents and Standards

Not Applicable to the SOP

11. Sample Collection, Preservation, Shipment and Storage

Not Applicable to the SOP

12. Quality Control

Not Applicable to the SOP

13. Calibration and Standardization

Not Applicable to the SOP

14. Procedure

14.1 Initial Calibration (ICAL), including Mass Spec tuning or instrument (GC) performance, if applicable

Check the following criteria:

14.1.1 Mass Spectrometer tuning criteria or other preliminary instrument performance check acceptable
14.1.2 Minimum number of calibration standards analyzed
14.1.3 Standards analyzed in appropriate time frame
14.1.4 Acceptance criteria met: statistics, monitoring compounds or internal standards
14.1.5 Calibration updated correctly: factors, retention times, correct data files used to update ICAL
14.1.6 Low standard at or near reporting limit
14.1.7 Linear range established, if applicable
14.1.8 ICV analysis acceptable: results, retention times, internal standards
14.1.9 Calibration Blank analysis acceptable, if applicable
14.1.10 Calibration Standards and Reagents properly identified (traceable)
14.1.11 Manual integrations acceptable, if applicable
14.1.12 All required information recorded in logbook or data file
14.1.13 For those analyses (e.g. GC/MS, GC, etc.) that generate separate report pages for the ICAL Standards, include the ICAL report pages with the ICAL Data Review Checklist.

14.2 Continuing Calibration Verification (CCV), including Mass Spec tuning or GC performance, if applicable

Check the following criteria:

14.2.1 Mass Spectrometer tuning criteria or other preliminary instrument performance check acceptable
14.2.2 CCV Acceptance criteria met: statistics, monitoring compounds, results, retention times, internal standards
14.2.3 Correct response or calibration factor from ICAL used for quantitation

14.2.4 Calculations performed accurately
14.2.5 Proper frequency of analysis
14.2.6 Calibration Blank analysis acceptable, if applicable
14.2.7 Calibration Standards and Reagents properly identified (traceable)
14.2.8 Manual integrations acceptable, if applicable
14.2.9 All required information recorded in logbook or data file
14.2.10 For those analyses (e.g. GC/MS, GC, Lachat, etc.) that generate separate report pages for the CCV and QC Samples, include the CCV and QC Sample report pages with the Batch Data Review Checklist.

14.3 Method Blanks (MB)

Check the following criteria:
14.3.1 Contamination level acceptable (< RL or other criteria)
14.3.2 Common laboratory contaminants level noted as acceptable, if applicable
14.3.3 False positives and false negatives
14.3.4 Proper frequency of analysis
14.3.5 Reagents properly identified (traceable)
14.3.6 Manual integrations acceptable, if applicable
14.3.7 All required information recorded in logbook or data file

14.4 Laboratory Control Samples (LCS/LCSD)

Check the following criteria:
14.4.1 Recovery and precision within acceptance limits
14.4.2 Calculations performed accurately
14.4.3 Proper frequency of analysis
14.4.4 Spiking Solution and Reagents properly identified (traceable)
14.4.5 Manual integrations acceptable, if applicable
14.4.6 All required information recorded in logbook or data file

14.5 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)

Check the following criteria:
14.5.1 Recovery and precision within acceptance limits
14.5.2 Calculations performed accurately
14.5.3 Proper frequency of analysis
14.5.4 Spiking Solution and Reagents properly identified (traceable)
14.5.5 Manual integrations acceptable, if applicable
14.5.6 All required information recorded in logbook or data file

14.6 Laboratory Duplicates, if applicable

Check the following criteria:
14.6.1 Recovery and precision within acceptance limits
14.6.2 Calculations performed accurately
14.6.3 Proper frequency of analysis
14.6.4 Manual integrations acceptable, if applicable
14.6.5 All required information recorded in logbook or data file

14.7 Surrogates

Check the following criteria:
14.7.1 Recovery within acceptance limits
14.7.2 Calculations performed accurately
14.7.3 Proper frequency of analysis (all standards, QC samples, and test samples spiked)
14.7.4 Spiking Solution and Reagents properly identified (traceable)
14.7.5 Manual integrations acceptable, if applicable
14.7.6 All required information recorded in logbook or data file

14.8 Internal Standards

Check the following criteria:
14.8.1 Recovery within acceptance limits
14.8.2 Calculations performed accurately
14.8.3 Proper frequency of analysis (all standards, QC samples, and test samples spiked)
14.8.4 Spiking Solution and Reagents properly identified (traceable)
14.8.5 Manual integrations acceptable, if applicable
14.8.6 All required information recorded in logbook or data file

14.9 Other Method specific QC samples (such as post digestion spikes)

Check the following criteria:
14.9.1 Recovery within acceptance limits
14.9.2 Calculations performed accurately
14.9.3 Proper frequency of analysis
14.9.4 Spiking Solution and Reagents properly identified (traceable)
14.9.5 Manual integrations acceptable, if applicable
14.9.6 All required information recorded in logbook or data file

14.10 Sample results

Check the following criteria:
14.10.1 Sample ID correct
14.10.2 Samples analyzed within appropriate time period (tune period)
14.10.3 Samples bracketed by acceptable calibration standards (CCVs)
14.10.4 False positives and false negatives
14.10.5 Sample results within instrument calibrated or linear range
14.10.6 Dilutions performed, if applicable
14.10.7 Sample results not within instrument calibrated or linear range and no dilution performed, flagged
14.10.8 Positive results confirmed (GC second column or GC/MS)
14.10.9 Appropriate units
14.10.10 Wet weight or dry weight basis for solids, if applicable
14.10.11 Manual integrations acceptable, if applicable
14.10.12 All required information recorded in logbook or data file
14.10.13 All sample results transcribed correctly to LIMS
14.10.14 Appropriate data flags used, if applicable

14.11 Sample dilutions, if applicable

Check the following criteria:
14.11.1 Sample ID correct
14.11.2 Sample diluted within calibrated or linear range (upper half of curve is preferred)
14.11.3 Calculations performed accurately
14.11.4 Manual integrations acceptable, if applicable
14.11.5 All required information recorded in logbook or data file

14.12 Manual calculations

Check the following criteria:
14.12.1 Calculations performed accurately
14.12.2 Solid sample results corrected for dry weight, if applicable
14.12.3 All required information recorded in logbook or data file
14.12.4 Secondary reviewer confirms 25% of the calculations by dating and initialing the logbook entry.

14.13 Manual integrations

Check the following criteria:
14.3.1 Manual integrations performed according to SOP
14.3.2 Record complete: hardcopy of "before" and "after" integrations
14.3.3 Initials and dates for both levels of review
14.3.4 Electronic data files for manually integrated peaks intact
14.3.5 All required information recorded in logbook or data file

14.14 Reporting Limits (RL)

Check the following criteria:
14.14.1 RL based on low ICAL standard or some other criteria
14.14.2 Appropriate units
14.14.3 RL elevated properly for diluted samples
14.14.4 RL elevated properly for solid samples reported as dry weight
14.14.5 RL elevated properly for samples with interferents, if applicable
14.14.6 All required information recorded in logbook or data file

14.15 Holding time criteria

Check the following criteria:
14.15.1 Samples analyzed within holding time period (hours to hours, days to days)
14.15.2 Samples not analyzed within holding time period, flagged
14.15.3 All required information recorded in logbook or data file

14.16 Sample preservation criteria

Check the following criteria:
14.16.1 Samples received with proper preservation (refrigeration, chemical as applicable)
14.16.2 Samples received without proper preservation, flagged
14.16.3 Samples preserved upon receipt at the laboratory, flagged

14.16.4 Preservation reagents properly identified (traceable), if applicable
14.16.5 All required information recorded in logbook or data file

14.17 Sample preparation criteria, including support equipment calibration

Check the following criteria:
14.17.1 Sample ID correct
14.17.2 Proper amount of sample was extracted or digested
14.17.3 Final extract or digestate volume appropriate
14.17.4 Support equipment properly calibrated and checked
14.17.5 Spiking Solutions and Reagents properly identified (traceable)
14.17.6 All required information recorded in logbook or data file
14.17.7 All sample results transcribed correctly to LIMS
14.17.8 Appropriate data flags used, if applicable

14.18 Sample storage criteria

Check the following criteria:
14.18.1 Samples stored under proper conditions
14.18.2 Samples not stored under proper conditions, flagged
14.18.3 All required information recorded in logbook or data file

14.19 Completeness of records

Check the following criteria:

14.19.1 Checklist completely filled out, all items checked
14.19.2 Signatures/initials and dates
14.19.3 Corrective actions noted
14.19.4 Corrections to hardcopy records done appropriately (no obliterations, no cross-outs, initials and date)
14.19.5 Corrections to electronic records done appropriately (changes recorded using analyst initials, password protection employed)
14.19.6 All required information recorded in logbook or data file

14.20 Corrective actions for QC failures

Check the following criteria:
14.20.1 Routine corrective actions noted, evidence of retesting, reanalysis, reprep
14.20.2 Non-Routine corrective actions noted, Corrective Action Report (CAR) filed
14.20.3 Quality Exception Report (QER) (if applicable), filed with data, job folder, QA Manager
14.20.4 All required information recorded in logbook or data file, and LIMS

14.21 Any additional comments or observations that would aid in the future reconstruction of the data

14.21.1 Note limited sample amount if not adequate for routine testing, elevated RL
14.21.2 Physical appearance of sample if non-routine
14.21.3 Appearance of digestate or extract if non-routine
14.21.4 Reference to reanalysis performed in a different sample batch
14.21.5 Replicate testing or confirmational testing performed to verify data

15. Data Reduction, Calculations and Loading

Not Applicable to the SOP

16. Method Performance

Not Applicable to the SOP

17. Pollution Prevention

Not Applicable to the SOP

18. Data Assessment and Criteria for Quality Control Measures

Not Applicable to the SOP

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the Data Review findings determine that the laboratory performance for a particular parameter is judged to be out of control, the problem must be immediately identified and corrected. The analytical results produced for that parameter are suspect. The time frame for the out of control situation will be determined. This will identify what client samples were analyzed during the time frame. Immediate corrective action includes written notification to clients that the data produced for the parameter may be affected and, if possible, to the degree that the data was affected.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data are revealed through the Data Review process, the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discuss options with the client. Reanalysis or reporting the data with qualification are alternatives. This may include the re-issue of amended reports with qualified data indicating that the previously reported results did not meeting the laboratory defined criteria. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting. For amended reports, the reason for the report amendment is clearly identified and explained.

Final data results must be qualified in the client report for results not meeting the laboratory-defined criteria.

The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and recorded by the Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21. Waste Management

Not Applicable to the SOP

22. References

22.1 STAT Analysis Corporation Quality Assurance Manual
22.2 SOP 230 Corrective Actions

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

Attachment 1 DATA REVIEW CHECKLIST - ICAL VOA GC/MS
Attachment 2 DATA REVIEW CHECKLIST - BATCH DATA VOA GC/MS
Attachment 3 DATA REVIEW CHECKLIST - ICAL SVOA GC/MS
Attachment 4 DATA REVIEW CHECKLIST - BATCH DATA SVOA GC/MS
Attachment 5 DATA REVIEW CHECKLIST - ICAL GC PESTICIDES AND PCB
Attachemtn 6 DATA REVIEW CHECKLIST - BATCH DATA GC PESTICIDES AND PCB
Attachment 7 DATA REVIEW CHECKLIST - ICAL and BATCH DATA ICP/MS
Attachment 8 DATA REVIEW CHECKLIST - ICAL and BATCH DATA MERCURY
Attachment 9 DATA REVIEW CHECKLIST - ICAL and BATCH DATA INORGANICS
Attachment 10 DATA REVIEW CHECKLIST - BATCH DATA ORGANICS
Attachment 11 DATA REVIEW CHECKLIST - ICAL DATA ORGANICS
Attachment 12 DATA REVIEW CHECKLIST - ICAL TO15A
Attachment 13 DATA REVIEW CHECKLIST - BATCH DATA TO15A
Attachment 14 DATA REVIEW CHECKLIST - ICAL Herbicides
Attachment 15 DATA REVIEW CHECKLIST - BATCH DATA Herbicides
Attachment 16 DATA REVIEW CHECKLIST - ICAL TO13A
Attachment 17 DATA REVIEW CHECKLIST - BATCH DATA TO13A

ATTACHMENT 1: DATA REVIEW CHECKLIST - ICAL

GC/MS Volatiles SOP 4000, EPA 8260B

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # /QER #
1	Tune meets criteria						
2	# of Stds _____ (minimum = 5)						
3	All Stds in tune period: 12hr All Stds. Sequenced w/in 36 hrs.						
4	Acceptance criteria met for: SPCCs: ≥0.1, ≥0.3 CCCs: ≤30% RSD %RSD ≤15% avg RF used r ≥0.99 linear regression used RRT ± 0.06 RT units						
5	Calibration updated: RF RT Mid-level used for int stds Correct data files used						
6	RL = low std = _____µg/L RL = _____ µg/L for (list)						
7	High std = _____µg/L High std = _____µg/L for						
8	ICV acceptable for: CCCs: ≤20%D SPCC's: ≥0.1, ≥0.3 RT: ± 30 seconds Int stds: -50% to +100% Other compounds						
9	Manual Integration acceptable: List files						
10	Tune & QC Solns ID recorded						
11	All other information recorded						

Additional Comments:_____

ATTACHMENT 2: DATA REVIEW CHECKLIST – BATCH DATA

GC/MS Volatiles SOP 4000, EPA 8260B

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	Tune meets criteria						
2	All Analyses in tune period 12hr						
3	CCV Acceptance criteria met: SPCCs: ≥0.1, ≥0.3 CCCs: ≤20%D RT: ± 30 seconds Int stds: -50% to +100% Correct ICAL RFs used						
4	Method Blank < RL False pos/neg checked Surrogates acceptable						
5	LCS/LCSD recovery acceptable Surrogates acceptable						
6	MS/MSD recov/RPD acceptable Surrogates acceptable						
7	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed						
8	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
9	Manual Integration acceptable: List files						
10	Int Stds Check (optional)						
11	Tune & QC Solns ID recorded						
12	All other information recorded						
13	LIMS transcription correct						
14	Analysis within holding time						
15	All samples preserved pH ≤2						
16	All samples stored properly						
17	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 3: DATA REVIEW CHECKLIST - ICAL

SemiVolatiles SOP 4020, EPA 8270C

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # / QER #
1	Tune meets criteria						
2	# of Stds (minimum = 5)						
3	All Stds in tune period: 12hr All Stds. Sequenced w/in 36 hrs.						
4	Acceptance criteria met for: SPCCs: ≥0.05 CCCs: ≤30 % RSD %RSD ≤15% avg RF used r ≥0.99 linear regression used RRT ± 0.06 RT units						
5	Calibration updated: RF RT Mid-level used for int stds Correct data files used						
6	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
7	High std = _____µg/mL High std = _____µg/mL for (list)						
8	ICV acceptable for: CCCs: ≤20%D RT: ± 30 seconds Int stds: -50% to +100% Other compounds						
9	Manual Integration acceptable: List files						
10	Tune & QC Solns ID recorded						
11	All other information recorded						

Additional Comments:_____

ATTACHMENT 4: DATA REVIEW CHECKLIST – BATCH DATA

SemiVolatiles SOP 4020, EPA 8270C

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Analysis date:_____ Batch Start Time:_____ Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	Tune meets criteria						
2	All Analyses in tune period 12hr						
3	CCV Acceptance criteria met: SPCCs: ≥0.05 CCCs: ≤20%D RT: ± 30 seconds Int stds: -50% to +100% Correct ICAL RFs used						
4	Method Blank < RL False pos/neg checked Surrogates acceptable						
5	LCS/LCSD recovery acceptable Surrogates acceptable						
6	MS/MSD recov/RPD acceptable Surrogates acceptable						
7	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed						
8	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
9	Manual Integration acceptable: List files						
10	Int Stds Check (optional)						
11	Tune & QC Solns ID recorded						
12	All other information recorded						
13	LIMS transcription correct						
14	Analysis within holding time						
15	All samples correct pH						
16	Autosampler tray checked						
17	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 5: DATA REVIEW CHECKLIST - ICAL

GC Pesticides and PCBs SOP 4050, EPA 8081A & 8082

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____Column:_____

ICAL Data File #s:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # / QER #
1	Breakdown meets criteria						
2	# of Stds_____(minimum = 5)						
3	All Stds in batch period: 12hr						
4	Acceptance criteria met for: %RSD ≤20% avg CF used r ≥0.99 linear regression used RT Windows established						
5	Calibration updated: CF RT Correct data files used						
6	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
7	High std = _____µg/mL High std = _____µg/mL for list						
8	ICV acceptable: ≤15%D (all) Or ≤20%D (average) List Comps ≥15%D RT: all comps in window						
9	Manual Integration acceptable: List files						
10	ICAL & QC Solns ID recorded						
11	All other information recorded						

Additional Comments:_____
_____
_____
_____

ATTACHMENT 6: DATA REVIEW CHECKLIST – BATCH DATA

GC Pesticides and PCBs SOP 4050, EPA 8081A & 8082

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____Column:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # QER#
1	Breakdown meets criteria						
2	All Analyses in 20 samples batch period						
3	CCV acceptable: ≤15%D (all) Or ≤20%D (average) List Comps ≥15%D RT: all comps in window						
4	Method Blank < RL False pos/neg checked Surrogates acceptable						
5	LCS/LCSD recovery acceptable Surrogates acceptable						
6	MS/MSD recov/RPD acceptable Surrogates acceptable						
7	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed Positive results confirmed						
8	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
9	Manual Integration acceptable: List files						
10	QC Solns ID recorded						
11	All other information recorded						
12	LIMS transcription correct						
13	Analysis within holding time						
14	Autosampler tray checked						
15	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 7: DATA REVIEW CHECKLIST – ICAL and BATCH DATA

Metals ICP/MS SOP 4510

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Analysis date:_____ Analysis Start Time:_____ Instrument:_____

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR# QER#
1	Tune Report: counts/RSD/oxides ++charge/background counts axis/width at 10%						
2	P/A factor adjustment performed						
3	Tune Check: RSDs, Mass Calibration and Resolution Check meet criteria						
4	# of Stds _____ (minimum = 3) Cal Blank acceptable						
5	Calibration: Correct data files used and printed, $r \geq 0.995$ on linear regression Int Stds pass						
6	ICAL & QC Solns ID recorded						
7	ICV acceptable: ≤10%D Int Stds						
8	ICB acceptable: <RL, Int Stds						
9	ICSA and ICSAB acceptable						
10	All CCV ≤ 10%D					(See attached)	
11	All CCB < RL					(See attached)	
12	Checked autosampler tray						
13	Method Blank < RL Int Stds acceptable						
14	LCS/LCSD recovery acceptable Int Stds acceptable						
15	MS/MSD recov/RPD acceptable Int Stds acceptable						
16	Samples: Correct Ids						
17	Samples: Correct Test Code						
18	Dilutions: >LDR, Int Std failure						
19	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
20	LIMS blank, spike, RPD ref correct						
21	LIMS prep batch information correct: weights, volumes						
22	LIMS transcription and calculations correct						
23	LIMS correct reporting units						
24	Analysis within holding time						
25	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 8: DATA REVIEW CHECKLIST – ICAL and BATCH DATA

Mercury SOP 4530, EPA 7470A & 7471A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # QER #
1	# of Stds_____ (minimum = 5)						
2	Cal Blank acceptable						
3	Acceptance criteria met r ≥0.995 linear regression						
4	Calibration updated						
5	RL = low std = _____µg/L						
6	High std = _____µg/L						
7	ICV acceptable: ≤10%D						
8	ICB acceptable: < RL						
9	All CCV ≤20%D						
10	All CCB < RL						
11	Method Blank < RL						
12	LCS/LCSD recovery acceptable						
13	MS/MSD recov/RPD acceptable						
14	Test Samples: Correct Ids RL, Units Results within curve Dilutions performed						
15	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
16	ICAL & QC Solns ID recorded						
17	All other information recorded						
18	LIMS transcription correct						
19	Analysis within holding time						
20	All samples preserved pH ‹ 2						
21	Autosampler tray checked						
22	Corrections properly recorded						

Additional Comments:_____

_____

_____

ATTACHMENT 9: DATA REVIEW CHECKLIST – ICAL and BATCH DATA

Inorganics Department

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Test Name:_____ SOP #:_____ Reference Method:_____

Analysis date:_____ Batch Start Time:_____ Instrument:_____

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER #
1	# of Stds _____ (minimum = 5)						
2	Cal Blank acceptable						
3	Acceptance criteria met r ≥0.995 linear regression						
4	Calibration updated: Correct data files used						
5	RL = low std = _____ mg/L						
6	High std = _____ mg/L						
7	ICV acceptable: ≤10%D						
8	ICB acceptable: < RL						
9	All CCV ≤10%D						
10	All CCB < RL						
11	Method Blank < RL						
12	LCS/LCSD recovery acceptable						
13	MS/MSD recov/RPD acceptable						
14	Lab Duplicate RPD acceptable						
15	Test Samples: Correct IDs RL, Units Results within curve Dilutions performed						
16	MB, LCS/LCSD, MS/MSD, or LD per batch of 20 samples						
17	ICAL & QC Solns ID recorded						
18	All other information recorded						
19	LIMS transcription correct						
20	Analysis within holding time						
21	All samples correct pH						
22	Autosampler tray checked						
23	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 10: DATA REVIEW CHECKLIST – BATCH DATA

Organics Department

Test:_____ SOP:_____

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Analysis date:_____ Batch Start Time:_____ Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	All Analyses bracket by CCV						
2	CCV Acceptance criteria met: CCCs: ≤15%D RT: ± 30 seconds Correct ICAL CFs used						
3	Method Blank < RL False pos/neg checked						
4	LCS/LCSD recovery acceptable						
5	MS/MSD recov/RPD acceptable						
6	Test Samples: Correct IDs RL, Units False pos/neg checked Results within curve Dilutions performed						
7	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
8	Manual Integration acceptable: List files						
9	QC Solns ID recorded						
10	All other information recorded						
11	LIMS transcription correct						
12	Analysis within holding time						
13	Autosampler tray checked						
14	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 11: DATA REVIEW CHECKLIST - ICAL

Organics Department

Test:_____ SOP:_____

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Analysis date:_____ Batch Start Time:_____ Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # / QER #
1	# of Stds_____ (minimum = 5)						
2	Acceptance criteria met for: %RSD ≤20% avg CF used r ≥0.99 linear regression used RRT ± 0.06 RT units						
3	Calibration updated: CF RT Correct data files used						
4	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
5	High std = _____µg/mL High std = _____µg/mL for (list)						
6	ICV acceptable for: CCCs: ≤15 %D RT: ± 30 seconds Other compounds						
7	Manual Integration acceptable: List files						
8	QC Solns ID recorded						
19	All other information recorded						

Additional Comments:_____

ATTACHMENT 12: DATA REVIEW CHECKLIST - ICAL

GC/MS Volatiles SOP 4010, EPA T015A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # /QER #
1	Tune meets criteria						
2	# of Stds _____ (minimum = 5)						
3	All Stds in tune period: 24 hr All Stds. Sequenced w/in 36 hrs.						
4	Acceptance criteria met for: CCC's: ≤30% RSD RRT ± 0.06 RT units %RSD ≤30% avg RF used r ≥0.99 linear regression used (list)						
5	Calibration updated: RF RT Mid-level used for int stds Correct data files used						
6	RL = low std _____ppbv RL = _____ ppbv for (list)						
7	High std = _____ppbv High std = _____ppbv for (list)						
8	ICV acceptable for: CCCs: ≤30%D RT: ± 30 seconds Int stds: 60-140% Other compounds						
9	Manual Integration acceptable: List files						
10	Tune & QC gas ID recorded						
11	All other information recorded						

Additional Comments:_____

ATTACHMENT 13: DATA REVIEW CHECKLIST – BATCH DATA

GC/MS Volatiles SOP 4010, EPA TO15A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # QER#
1	Tune meets criteria						
2	All Analyses in tune period 24hr						
3	CCV Acceptance criteria met: CCCs: ≤30%D RT: ± 30 seconds Int stds: 60 to 140 % Correct ICAL RFs used						
4	Method Blank < RL False pos/neg checked						
5	LCS/LCSD %R = 70-130 %RSD < 25%						
6	Test Samples: Correct IDs False pos/neg checked RL, Units Results within curve Dilutions performed						
8	MB, LCS/LCSD per batch of 20 samples						
9	Manual Integration acceptable: List files						
10	Int Stds Check (optional)						
11	Tune & QC gas ID recorded						
12	All other information recorded						
13	LIMS transcription correct						
14	Analysis within holding time						
15	Canister position on Unity verified						
16	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 14: DATA REVIEW CHECKLIST - ICAL

Herbicides SOP 4090, EPA 8321A

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Analysis date:_____ Batch Start Time:_____ Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # / QER #
1	# of Stds _____ (minimum = 5)						
2	Acceptance criteria met for: CCC: ≤20 % RSD %RSD ≤ 20 % avg CF used r ≥0.99 linear regression used						
3	Calibration updated: CF RT Correct data files used						
4	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
5	High std = _____µg/mL High std = _____µg/mL for (list)						
6	ICV acceptable for: % Diff: ≤20%D RT: ± 30 seconds Other compounds						
7	Manual Integration acceptable: List files						
8	QC Solns ID recorded						
9	All other information recorded						

Additional Comments:_____

ATTACHMENT 15: DATA REVIEW CHECKLIST – BATCH DATA

Herbicides SOP 4090, EPA 8321A

First Level Reviewer:_____ Review Date:_____

Second Level Reviewer:_____ Review Date:_____

Analysis date:_____ Batch Start Time:_____ Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	All Analyses in 20 sample batch period						
2	CCV Acceptance criteria met: % Diff: ≤15%D RT: ± 30 seconds Correct ICAL CFs used						
3	Method Blank < RL False pos/neg checked Surrogate acceptable						
4	LCS/LCSD recovery acceptable Surrogate acceptable						
5	MS/MSD recov/RPD acceptable Surrogates acceptable						
6	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed						
7	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
8	Manual Integration acceptable: List files						
9	QC Solns ID recorded						
10	All other information recorded						
11	LIMS transcription correct						
12	Analysis within holding time						
13	All samples correct pH						
14	Autosampler tray checked						
15	Corrections properly recorded						

Additional Comments:_____

ATTACHMENT 16: DATA REVIEW CHECKLIST - ICAL

SemiVolatiles SOP 4030, EPA TO-13A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # / QER #
1	Tune meets criteria						
2	# of Stds _____ (minimum = 5)						
3	All Stds in tune period: 12hr						
4	Acceptance criteria met for: CCCs: ≤30 % RSD RRT ± 0.06 RT units						
5	Calibration updated: RF RT Mid-level used for int stds Correct data files used						
6	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
7	High std = _____µg/mL High std = _____µg/mL for (list)						
8	ICV acceptable for: CCCs: ≤30%D RT: ± 30 seconds Int stds: -50% to +100% Other compounds						
9	Manual Integration acceptable: List files						
10	Tune & QC Solns ID recorded						
11	All other information recorded						

Additional Comments:_____

ATTACHMENT 17: DATA REVIEW CHECKLIST – BATCH DATA

SemiVolatiles SOP 4030, EPA TO-13A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	Tune meets criteria						
2	All Analyses in tune period 12hr						
3	CCV Acceptance criteria met: CCCs: ≤30%D RT: ± 20 seconds Int stds: -50% to +100% Correct ICAL RFs used						
4	Method Blank < RL False pos/neg checked Surrogates acceptable						
5	LCS/LCSD recovery acceptable Surrogates acceptable						
6	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed						
7	MB, LCS/LCSD per batch of 20 samples						
8	Manual Integration acceptable: List files						
9	Int Stds Check (optional)						
10	Tune & QC Solns ID recorded						
11	All other information recorded						
12	LIMS transcription correct						
13	Analysis within holding time						
14	Autosampler tray checked						
15	Corrections properly recorded						

Additional Comments:_____

SOP ADDENDUM

SOP No. & TITLE: SOP 1250 DATA REVIEW
Revision 01, Effective Date: 11/17/05

Issued by Laurie Fetterman, QA Manager

Approved by: Dennis Jachim, Technical Manager

Date of Issue: May 19, 2005

Section 23 (additions are *italicized*, ~~deletions are strikethroughs~~)
See Attached Sheets

The initials/signature of the QA Manager and the Technical Manager indicate that this is a controlled document.

ATTACHMENT 5: DATA REVIEW CHECKLIST - ICAL

GC Pesticides and PCBs SOP 4050, EPA 8081A & 8082

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____Column:_____

ICAL Data File #s:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR #/ QER#
1	Breakdown meets criteria						
2	# of Stds____ (minimum = 5)						
3	All Stds in batch period: 12hr						
4	Acceptance criteria met for: %RSD ≤ 20% avg CF used r ≥0.99 linear regression used RT Windows established						
5	Calibration updated: CF RT Correct data files used						
6	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
7	High std = _____µg/mL High std = _____µg/mL for list						
8	ICV acceptable: ≤15%D (all) Or ≤20 15%D (average) List Comps > 15%D RT: all comps in window						
9	Manual Integration acceptable: List files						
10	ICAL & QC Solns ID recorded						
11	All other information recorded						

Additional Comments:_____
_____
_____
_____

ATTACHMENT 6: DATA REVIEW CHECKLIST – BATCH DATA

GC Pesticides and PCBs SOP 4050, EPA 8081A & 8082

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____Column:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	Breakdown meets criteria						
2	All Analyses in 20 samples batch period						
3	CCV acceptable: ≤15%D (all) Or ≤20 15%D (average) List Comps > 15%D RT: all comps in window						
4	Method Blank < RL False pos/neg checked Surrogates acceptable						
5	LCS/LCSD recovery acceptable Surrogates acceptable						
6	MS/MSD recov/RPD acceptable Surrogates acceptable						
7	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed Positive results confirmed						
8	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
9	Manual Integration acceptable: List files						
10	QC Solns ID recorded						
11	All other information recorded						
12	LIMS transcription correct						
13	Analysis within holding time						
14	Autosampler tray checked						
15	Corrections properly recorded						

Additional Comments:_____

SOP ADDENDUM

SOP No. & TITLE: SOP 1250 DATA REVIEW
Revision 01, Effective Date: 11/17/05

Issued by Laurie Fetterman, QA Manager

Approved by: Dennis Jachim, Technical Manager

Date of Issue: August 17, 2005

Section 23 (additions are *italicized*, ~~deletions are strikethroughs~~)
See Attached Sheets

The initials/signature of the QA Manager and the Technical Manager indicate that this is a controlled document.

ATTACHMENT 14: DATA REVIEW CHECKLIST - ICAL

Herbicides SOP ~~4090~~ 4080, EPA 8321A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____
ICAL Data File #s:_____

Page 1 of 1 Initial Calibration (ICAL)

Item	Aspect	1st Yes	Level No	2nd Yes	Level No	Comments: record routine corrective actions here	CAR # / QER #
1	# of Stds _____ (minimum = 5)						
2	Acceptance criteria met for: CCC: ≤20 % RSD %RSD ≤ 20 % avg CF used r ≥0.99 linear regression used						
3	Calibration updated: CF RT Correct data files used						
4	RL = low std = _____µg/mL RL = _____ µg/mL for (list)						
5	High std = _____µg/mL High std = _____µg/mL for (list)						
6	ICV acceptable for: % Diff: ~~≤20~~ *15*%D RT: ± 30 seconds Other compounds						
7	Manual Integration acceptable: List files						
8	QC Solns ID recorded						
9	All other information recorded						

Additional Comments:_____

ATTACHMENT 15: DATA REVIEW CHECKLIST – BATCH DATA

Herbicides SOP ~~4090~~ 4080, EPA 8321A

First Level Reviewer:_____Review Date:_____

Second Level Reviewer:_____Review Date:_____

Analysis date:_____ Batch Start Time:_____Instrument:_____

Page 1 of 1 Continuing Calibration Verification (CCV) and Batch Data

Item	Aspect	1st Level Yes	1st Level No	2nd Level Yes	2nd Level No	Comments: record routine corrective actions here	CAR # QER#
1	All Analyses in 20 sample batch period						
2	CCV Acceptance criteria met: % Diff: ≤15%D RT: ± 30 seconds Correct ICAL CFs used						
3	Method Blank < RL False pos/neg checked Surrogate acceptable						
4	LCS/LCSD recovery acceptable Surrogate acceptable						
5	MS/MSD recov/RPD acceptable Surrogates acceptable						
6	Test Samples: Correct IDs Surrogates acceptable False pos/neg checked RL, Units Results within curve Dilutions performed						
7	MB, LCS/LCSD, MS/MSD per batch of 20 samples						
8	Manual Integration acceptable: List files						
9	QC Solns ID recorded						
10	All other information recorded						
11	LIMS transcription correct						
12	Analysis within holding time						
13	All samples correct pH						
14	Autosampler tray checked						
15	Corrections properly recorded						

Additional Comments:_____

SOP ADDENDUM

SOP No. & TITLE: 1250 Data Review
Revision Effective Date: November 22, 2006

Issued by: Pinaki Banerjee, QA Director

Approved by: Dennis Jachim, Technical Manager

Date of Issue: November 22, 2006

Section (additions are *italicized*, ~~deletions are strikethroughs~~)

Section 5

The review is recorded by the secondary reviewer signing and dating a page of a notebook or by the use of checklists customized for each area of the laboratory and its particular test methodology. Each person performing the first level review and the second level review is required to sign his name or initials and record the date of the review on the checklist. The third level of review is not part of the data review checklist. The signature of the Project Manager or Laboratory Director on the final report affirms his review of the data. All data must be reviewed in this manner prior to release.

If the review reveals a deficiency in any area of data production, the secondary reviewer will return the notebook or the data packet to the analyst with the appropriate comments to take corrective action such as re-analysis, reintegration, or additional review.

For Microbiology and asbestos by Phase Contrast Microscopy (PCM), the analyst reviews the data initially and all data entries checked 100% and then the data undergoes a second review by a technical peer or supervisor. Secondary review is documented by signing on the space "QC by" on the STAT chain of custody or the asbestos analysis count sheet.

For Microbiology, the analyst rereads one out of 20 samples. If there is a discrepancy in results, a second analyst analyzes the sample to verify results. One out of 20 samples are re-read by a different analyst. Reference slides and texts are utilized in the verification process, ensuring that the morphological details match. Intra- and interanalyst QC data are documented in logbooks and also on the network under microbiology in excel spreadsheets.

The QA Manager, as part of the Internal QA Audit process, reviews 5% of the data produced in the laboratory. One of the tools used in this audit process is the data review checklist. When deficiencies are noted by the QA manager during his internal audit of data batches, the corporation's corrective action process is employed to remedy these situations. The appropriate Department Manager is asked to take immediate corrective action and to implement the necessary changes in an expedient manner. Additional audits and follow up audits are performed as necessary to verify that corrective actions have been implemented and are successful in correcting the respective deficiencies.

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 1400

LIMS

Revision 00
Effective Date: June 12, 2003

Author: **Dennis Jachim**

Printed Name	Signature/Date
Dennis Jachim **Technical Manager**	_____
Ian H. Graske **QA Manager**	_____
Thomas M. Bauer **Laboratory Director**	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

Copy Number: _____

The absence of a Copy Number indicates this is an uncontrolled copy of the document supplied for information only.

Table of Contents

1. Identification of Test Method

SOP Title: LIMS

2. Applicable Matrix or Matrices

Not Applicable to the SOP

3. Detection Limits

Not Applicable to the SOP

4. Scope and Application

This SOP details the procedures used by laboratory personnel to operate the Laboratory Information Management System (LIMS). The LIMS is used to record information that encompasses the laboratory's production of data related to individual test method batches, to ancillary support services, and to sustaining records of sample condition. The information may be sorted, tracked, and organized to allow for operational review to ensure that the information entered has been assessed for quality, accuracy, and completeness.

The function of the LIMS is appropriate and effective to the current level of laboratory activity. Additional utilities in the LIMS program are available but are not in use as of the effective date of this SOP. These additional utilities are not addressed in this SOP.

5. Summary of Test Method

The LIMS is accessed, via any PC located on the computer network, by individual laboratory personnel using their unique log-in ID and password. Individuals, using separate keystrokes, enter information into LIMS or large blocks of data may be imported from an independent software program associated with analytical instrumentation analysis (direct upload). Once entered, information in the LIMS may be reviewed and edited. Edits are tracked using that person's unique account ID. After final review, the information is "locked" in the system. The information may be reviewed by anyone who has access to the LIMS, but only a limited number of management personnel may edit the information at this time. Again, any changes made to the stored information are tracked and identified by the person making the change.

The information stored in LIMS is used to generate a variety of reports. These include: Test Data Reports, Case Narratives, Quality Control Data Reports, Sample Condition Reports, Work Order Summary, Backlog Reports, Invoices, and Corrective Action Reports.

The functions of the LIMS and the procedures used by the laboratory personnel to operate the LIMS are detailed in Section 14 of this SOP.

If any review of the information in the LIMS reveals a deficiency in any area of data entry, the person performing the review has the following options: (1) correct the information and record the change, (2) make a request to the person who initially entered the information to correct same, or (3) take steps to initiate a corrective action dependent upon the severity of the problem associated with the incorrect entry.

The QA Manager, as part of the Internal QA Audit process, reviews 5% of the data produced in the laboratory. Part of this review encompasses the information entered into LIMS. When deficiencies are noted by the QA Manager during his internal audit, the corporation's corrective action process is employed to remedy these situations. The appropriate Department Manager must take immediate corrective action and implement the necessary changes in an expedient manner. Additional audits and follow up audits are performed as necessary to verify that corrective actions have been implemented and are successful in correcting the respective deficiencies.

Additional documentation is through the use of the Corrective Action Report (STAT SOP 230 Corrective Action).

6. Definitions

Laboratory Information Management System (LIMS) - a software program, OMEGA ME version ELIMS 8.0 purchased from Khemia, that is used in the day to day operation of the laboratory to record and track information pertaining to sample management, sample analysis, and the supporting data associated with these tasks. This program is administered and maintained by the laboratory's Technical Manager.

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

Not Applicable to the SOP

8. Safety

Not Applicable to the SOP

9. Equipment and Supplies

Not Applicable to the SOP

10. Reagents and Standards

Not Applicable to the SOP

11. Sample Collection, Preservation, Shipment and Storage

Not Applicable to the SOP

12. Quality Control

Not Applicable to the SOP

13. Calibration and Standardization

Not Applicable to the SOP

14. Procedure

14.1 Initial Access to the LIMS

14.1.1 From the PC windows desktop, double click on the STAT _ME (OMEGA) icon

14.1.2 Enter user ID and password (DO NOT SHARE THIS INFORMATION WITH OTHER EMPLOYEES). This is required in order to track the individual entering or changing information.

14.1.3 The main screen should display OMEGA ME and an initial list of choices (See Attachment 1).

14.1.4 There are 8 "Large Buttons" listed. Details on the function and use of each of these large buttons are addressed in the following sections of this SOP.

- BackLog Rpt: see section 14.2
- Prep: see section 14.3
- WorkOrders: see section 14.4
- Connect to Backend: NOT IN USE
- Sample Tracker: NOT IN USE
- Data Entry: see section 14.5
- Reports: see section 14.6
- Quit: used to exit the LIMS and return to the windows desktop

14.1.5 "Categories" section: This block contains 10 categories as listed: Details on the function and use of each of these categories are addressed in the following sections of this SOP.

- System Administration: see section 14.7
- LIMS Configuration: see section 14.8
- Laboratory Management: see section 14.9
- Coordination: see section 14.10
- Analytical: see section 14.11
- Test Information: see section 14.12
- Sales: see section 14.13
- Quality Control: see section 14.14
- Reporting: see section 14.15
- Operations: see section 14.16

Click on the radio button for an individual "Category" and the sub-sections for that category are listed to the right in the field labeled "Options." Click on the option of choice. NOTE: some of the associated options in certain categories are not currently in use.

14.1.6 When finished with information entry or review, return to the main screen and click on **QUIT** to exit LIMS. DO NOT stay logged into LIMS if you are leaving the workstation. Another individual may enter information, but the entry (or change) will be registered to your account. In addition, the daily backup of the database is incomplete if employees are remain logged on overnight.

14.2 BackLog Rpt

The Backlog report provides the analyst with useful information concerning in-house work (See Attachment 2). Information such as client, sample number, collection date, holding time, test code, select list data, client-specified matrix spike, test due date, login review status, and storage location can be viewed on the report.

14.2.1. Click on the drop down list for **Test Code** to view all in-house work for ONLY that test code.

14.2.2. Select a department to view all in-house work for that particular department.

14.2.3. Click on the drop down list under the **Group By** to select the order in which to view the samples on the Backlog report.

14.2.3.1 Priority – This orders the Backlog report by test due date or by holding time, whichever comes first.

14.2.3.2 HoldingTime – This orders the Backlog report by the holding time.

14.2.3.3 TestDueDate – This orders the Backlog report by the test due date.

14.2.3.4 SampID – This orders the Backlog report by the individual sample number.

14.2.3.5 TestCode – This orders the Backlog report alphabetically by the LIMS test code.

14.2.4. Select **Preview Report** button to view the Backlog report on screen.

Note: **Preview Report** button only allows one department or one test code Backlog report to be previewed on screen.

14.2.5. Select **Generate Report** button to print a hardcopy of the BackLog report. Multiple department BackLogs may be printed.

14.2.6. The **Missing Analytes** report allows the analyst to view missing compounds or analytes on samples.

14.2.7 The **View Select** button brings up another form. Selecting a test code and sample, allows the analyst to view the analytes or compounds that need to be reported on the sample.

14.2.8 Select **Close** button to close the BackLog Report form.

14.3 Prep

This screen allows analysts to enter and review prep information for samples (See Attachment 3).

Note: The "Reagents/Spikes" tab allows analysts to track reagents and spikes used in the preparation process. This function is currently not being used.

Adding Prep Batches:

14.3.1. Click on the **Add** button on the toolbar.

14.3.2. Select a "Technician" from the drop down list, and change the "Start Date". The Start Date should include a date and time.

14.3.3. Select a "Prep Code" from the drop down list. LIMS will automatically assign a new batch number.

14.3.4. Click **LoadSamps**. Select HoldTime or DueDate, and select **Auto** or **User**. **Auto** will automatically add samples to the batch based on HoldTime or DueDate. **User** allows the analysts to select which samples are included in the prep batch

14.3.5. Selecting **User** brings up a Sample Selection Box. Select the available samples from the left. Use the single lined arrow to select (or deselect) one sample at a time. Use the double lined arrow to select (or deselect) all the samples. When the appropriate samples have been selected, click **OK**.

14.3.6. Fill in initial sample size, and final volume, as appropriate.

14.3.7. Enter an "End Date", hit enter, and when prompted to save the batch, click **Yes**. The prep information has now been recorded into LIMS.

14.3.8. Click **Labels** to automatically generate prep labels for the sample. Select the available samples from the batch on the left. Use the single lined arrow to select (or deselect) one sample at a time. Use the double lined arrow to select (or deselect) all the samples in the batch. Clicking **OK** will print preview the sample labels. The prep label contains the following information: Sample Number, Prep Code, Batch Number, and Prep Date.

14.3.9. Click **Print** to print preview a Prep Batch Report.

Reviewing Prep Batches:

14.3.10. Select "Open", "Closed", or "All" to select an open batch, closed batch, or both.

14.3.11. Select a Prep Code from the "Filter By" drop down list.

14.3.12. Click the appropriate batch number from the list. This brings up the Prep Batch for review.

14.4 WorkOrders

Project Management functions are accessed on this form (See Attachment 4). Project management can view the status of the workorder including analytical results, sample test status, case narratives, and billing/invoicing information. See SOP 300 Sample Receipt and Login, Section 14.19, on creating workorders and logging in samples.

14.4.1 The "Main" tab is for viewing client, project, and general workorder information.

Note: **Export Login Info**, **Import Data from Sub**, **Import Login from**, and **Export Data to Prime** can transfer data readily between laboratories working with this LIMS system. These functions are not currently being used.

14.4.2 Click Select All  to select all the workorders.

14.4.3 Click **LoginReview** to view all workorders waiting to be login reviewed.

14.4.4 Click **Completed** to view all workorders that have been login reviewed but are not completed.

14.4.5 Click **Validated** to view all workorders that have been login reviewed and completed, but are not validated.

14.4.6 Click **Reported** to view all workorders that have been login reviewed, completed, and validated, but are not reported.

14.4.7 Click **Invoiced** to view all workorders that have been login reviewed, completed, validated, and reported, but are not invoiced.

14.4.8 Click **Find IT** to selectively query the workorders by workorder number, Client ID, Project ID, date received, or by the Client Sample ID. Click **ClearGrid** to remove the filters selected. Click **Run Query** to query the workorders based on the filters selected.

14.4.9 The "ReportOptions" tab is for viewing and editing analytical data report options, and invoicing address information.

14.4.10 The "InvoiceInfo" tab allows viewing of test pricing, and editing invoice totals and client purchase order number.

Note: Individual test pricing must be changed in Login.

14.4.11 The "Narrative" tab is for applying and reviewing case narratives applicable to the samples or sample analysis for the workorder.

14.4.12 The "Sample Test Status" gives a status on the tests for the workorder.

14.4.13 The "Analytical Results" tab is for viewing sample analytical results.

14.4.14 Click **CopyWO** to copy a previous workorder and its information into the current workorder.

14.4.15 Click **Coolers** to track laboratory coolers (currently not being used).

14.4.16 Click **ChkList** to generate a Sample Receipt Checklist Report.

14.4.17 Click **WO COC** to generate a chain of custody for the workorder and the requested analyses.

14.4.18 Click **WO Rpt** to generate a Workorder Sample Summary Report.

14.4.19 Click **DupData** to view duplicate analytical data within LIMS (See Sect. 14.10.11)

14.4.20 **Reports** button (see Section 14.6)

14.4.21 Click **Login** to review, edit, and modify sample login information. See SOP 300 Sample Receipt and Login, Section 14.19, on logging in samples.

14.5 Data Entry

Data Entry has two distinct forms associated with it. The first form (Analytical Runs) shown in Attachment 5 gives general information on the individual samples. Clicking the ***Data*** button will bring the form (Data Entry) shown in Attachment 6. This brings up analytical data for samples listed on the Analytical Runs form.

Analytical Runs – Information concerning the Sample Number, Test Code, Sample Type, prep information, analysis date/time, percent moisture, dilution factor, and significant figures to report are stored here.

14.5.1. Click Select All  to select all the Analytical Runs.

14.5.2. Filter the Analytical Runs by selecting an instrument from the drop down list, and/or selecting an individual analyst from the drop down list. The index displays the filtered records.

14.5.3. The "Main" page of the Analytical runs displays information on the instrument used, analyst, and the run start date. The Ical and column ID are currently not being used.

14.5.4. The "CAL Stds" page allows the analyst to select standards used in the analysis that have been entered into LIMS (See Sect. 14.11.7). This is currently not being used.

14.5.5. The **Composite**, **Back Fracs**, **Dual Col/Det**, and **CLP Links** buttons are currently not being used, so their function will not be described.

14.5.6. Click **QA Auth** to enter a QA password to validate or unvalidate individual samples. Enter your QA password and click **OK**. Click **Cancel** to close the form. Only validated records can be reported.

14.5.7. Click **QA Seq** to validate all samples in the Analytical Run. Enter your QA password and click **OK**. Click **Cancel** to close the form. Only validated records can be reported.

14.5.8. Click **Re-Extract** to select samples to be re-prepared and re-analyzed. Select the available samples on the left. Use the single lined arrow to select (or deselect) one sample at a time. Use the double lined arrow to select (or deselect) all the samples in the analytical run. Clicking **OK** will return the samples to the prep analyst BackLog. Click **Close** to close the form.

14.5.9. Click **Load Samps** to manually add samples to the Analytical Run. Samples are selected by WORK ORDER and TEST CODE or can include multiple Work Orders by selecting a test code only. Selecting the appropriate information will display a listing of available samples. Only samples that have been Log-In Reviewed will be displayed. Use the single lined arrow to select (or deselect) one sample at a time. Use the double lined arrow to select (or deselect) all available samples. Clicking **OK** will load the samples into the Analytical Run. Click **Close** to close the form.

14.5.10. Click **Data Import** to automatically import samples into the analytical run. Select an import specification from the drop down list, click **RUN IMPORT**, and select the file(s) to import.

14.5.11. Click **Import Specs** to show a form listing the various import specifications that the LIMS uses to automatically read and import data. A listing of the different import specifications is displayed on the left. The file type that LIMS will be reading, and information on where LIMS can find the required information is displayed. Clicking **Header / Field Info** allows the user to add or change this information. Contact your Department Manager or the Technical Manger to determine which specification to use to automatically import data. *This should never be changed, except by a Department Manager or the Technical Manager.*

14.5.12. Click **Export to Excel** to export samples to Excel. This allows for the exporting of existing samples/tests into an Omega Excel File for adding of data and back importing into Analytical Runs. Samples are selected by WORK ORDER and TEST CODE or can include multiple Work Orders by selecting a test code only. Samples can also be selected by Prep Batch. Samples selected by Prep Batch do not need a WorkOrder or TestCode. Select the necessary information, click  , and select the file to accept the samples. Click  to close the form.

14.5.13. Click **Update Pmoist** to convert previously imported sample results to a dry weight basis. This only works on Analytical Runs that include PMOIST as a Test Code, and if the samples have been validated.

14.5.14. Click ***Data*** to view the analytical data for the samples.

Data Entry – Information concerning the analyte type, Transmit (reported or non-reported), analyte, reporting units, raw value, calculated value, final reporting value, spike levels, qualifiers, MDL, PQL, % recovery, and % RPD are all stored here. A list of samples from the Analytical Run is displayed under the index on the left. Select a sample to view its data.

14.5.15. Click **Xcalc** to calculate certain parameters for organic analyses. This is currently not being used.

14.5.16. Click **Tics** to import Tentatively Identified Compounds for that sample. Select an import specification and click **OK**. Click **Close** to close the form. This is currently not being used.

14.5.17. Click **TRANS** to filter and display only analytes to be reported (records displayed Y under the "T" column). The button then changes to **ALL**. Click **ALL** to display all analytes.

14.5.18. Select a sample from the drop down list to view the sample. This is the same as selecting the sample from the index at the left.

14.5.19. Click **TOGGLE** to report (or not report) an analyte for selected samples and/or Test Codes for the Analytical Run. This is currently not being used.

14.5.20. Click **QA Auth** to enter a QA password to validate or unvalidate individual samples. Enter your QA password and click **OK**. Click **Cancel** to close the form.

14.5.21. Click **Calc Samp** to calculate final results for the individual sample. Click **Yes** to run through the calculation queries. Click **No** to cancel the calculation. LIMS utilizes all significant figures it receives for its calculations, and then rounds the number for final reporting.

14.5.22. Click **Calc SEQ** to calculate final results for the entire Analytical Run. Click **Yes** to run through the calculation queries. Click **No** to cancel the calculation. LIMS utilizes all significant figures it receives for its calculations, and then rounds the number for final reporting.

14.5.23. Click **Rslts** to view analytical results for all samples in the Analytical Run. The sample number and sequence number are listed across the top, analytes are listed down, with analytical results placed accordingly.

14.5.24. Click **Run** to preview an analytical run summary report listing the sequence number, sample number, file number, and date/time analyzed. This is only on the GCMS Volatiles computers to aid in the data review process.

14.5.25. Click **Petra** to preview an analytical summary report for the individual sample. This is only on the GCMS Volatiles computers to aid in the data review process.

Importing Data – The following outline procedures used to import data into LIMS. Generally, the data import is either manual or automatic. Certain analyses undergo a semi-automated data entry. For example, the Cetac Mercury Analyzer generates a file that is formatted (See Sect. 14.11.10), and the

formatted file is automatically imported. Contact your Department Manager or the Technical Manager for exact specifications.

14.5.26. Click **Add** on the toolbar to add an Analytical Run.

14.5.27. Select an Instrument from the drop down list.

14.5.28. Enter a Run start date. LIMS automatically generates a unique Analytical Run.

14.5.29. Select the Analyst for the Analytical Run. LIMS will only accept one analyst.

14.5.30. Automatic Data Entry

14.5.30.1 Click **Data Import** to automatically import samples into the analytical run. Select an import specification from the drop down list, click **RUN IMPORT**, and select the file(s) to import.

14.5.31. Manual Data Entry – 3 different options

14.5.31.1 Click **Load Samps** to manually add samples to the Analytical Run. Samples are selected by WORK ORDER and TEST CODE or can include multiple Work Orders by selecting a test code only. Selecting the appropriate information will display a listing of available samples. Only samples that have been Login Reviewed will be displayed. Use the single lined arrow to select (or deselect) one sample at a time. Use the double lined arrow to select (or deselect) all available samples. Clicking **OK** will load the samples into the Analytical Run. Click **Close** to close the form. Analytical data for these samples must then be entered manually under Data Entry.

14.5.31.2 Click **Export to Excel** to export samples to Excel. Samples are selected by WORK ORDER and TEST CODE or can include multiple Work Orders by selecting a test code only. Samples can also be selected by Prep Batch. Samples selected by Prep Batch do not need a WorkOrder or TestCode. Select the necessary information, click  , and select the file to accept the samples. Enter analytical data into the spreadsheet, and import the spreadsheet according to Sect. 14.5.30.1.

14.5.31.3 Manually type the Sample Number into the Analytical Run, type in the Test Code, and add the Sample Type (SAMP). Analytical data for these samples must then be entered manually under Data Entry.

14.5.32. Verify BLKRef (sequence number corresponding to the method blank for the batch), SPKRef (sequence number corresponding to the method blank for LCS or sequence number corresponding to the original sample number for MS(D)), and RPDRef (sequence number corresponding to the MS for MSD sample or sequence number corresponding to the original sample number for DUP analysis) numbers.

14.5.33. Verify prep information by viewing the BatchID, prep date, and PFac columns.

14.5.34. Verify the dilution factor under the DF column.

14.5.35. Click ***Data*** to enter the Data Entry screen.

14.5.36. Click **Calc Samp** to calculate final results for an individual sample. Click **Yes** to run through the calculation queries.

14.5.37. Click **Calc SEQ** to calculate final results for the entire Analytical Run. Click **Yes** to run through the calculation queries.

14.5.38. Review data for acceptability.

14.5.39. Click  to close the Data Entry screen.

14.6 Reports

Analytical Data Reports, Invoices, and QC Reports are generated (See Attachment 7). Selecting **Base Report** shows the first form in Attachment 7. Selecting **QC Report** shows the second form in Attachment 7. Selecting **Both** allows a combination of the two forms in Attachment 7.

Analytical Data Reports and Invoice Reports – Analytical Reports can be generated by WorkOrder, Sample, individual Department, or by individual TestCodes. Invoice Reports are only generated by WorkOrder.

14.6.1 Select the appropriate WorkOrder from the drop down list. A list of TestCodes and Departments applicable to that Workorder will appear.

14.6.2 Select an individual Sample from the drop down list, as needed.

14.6.3 Select a Department or specific TestCode, as needed.

14.6.4 Check the box next to Cover Letter, and select a Cover Letter from the drop down list, as needed. See Section 14.8.8 for additional information.

14.6.5 Check the box next to Sample Summary to include a Sample Summary Report.

14.6.6 Check the box next to Case Narrative to include a case narrative report.

14.6.7 Check the box next to Analytical Reports, and select the appropriate report type from the drop down list. See below for the most common report types.

14.6.7.1 Base Report – A report with individual sample fractions per report page.

14.6.7.2 Base Report Consolidated – A report that consolidates the sample fractions into one reporting sample per report page.

14.6.7.3 Base Report Continuous – A report with more than one sample per report page.

14.6.8 Select the options for the analytical report by selecting the radio buttons. J-Qualifiers will add to the report any result detected above the Method Detection Limit but below the Reporting Limit. Surrogates will add Surrogate recoveries to the analytical report. TICS will add any Tentatively Identified Compounds to the analytical report (not used).

14.6.9 Select **Final** if it is a final analytical report. Select **Prelim** if it is a preliminary report.

14.6.10 Check the box next to Dates Report to include the Dates Report.

14.6.11 Check the box next to Invoice to include an Invoice Report. See Section 14.4.3 for additional information.

14.6.12 **Select All** will select all the check boxes, and **Clear All** will clear all the check boxes.

14.6.13 **In-House Report** will preview the report selected. Note: For In-House Reports, the tests do not have to be validated. **Preview Report** will preview the report selected. **Generate Report** will print the selected reports. **Close** will close the form.

QC Analytical Reports – QC Analytical Reports are generated by WorkOrder and by Department.

14.6.14 Select the appropriate WorkOrder from the drop down list. A list of TestCodes and Departments applicable to that Workorder will appear.

14.6.15 Select the Departments to report, as needed.

14.6.16 Select the appropriate SampTypes to report (sample batch information).

14.6.17 Clicking the radio button by Cal Types allows SampTypes for instrument QC to be included.

14.6.18 If needed, select a specific QC batch number from the Print by QC Batch drop down list.

14.6.19 Select how to associate the QC: **TestCode**; **TestNo**; or **Batch**. Batch is the default.

14.6.20 Select the Options for reporting: Report Surrogates will show surrogate recoveries on the QC Analytical Report. Limit Analytes to SEL List Only will report only the analytes that were reported on the Analytical Data Report.

14.6.21 Special Report drop down list gives one option: QCSurrReport. Note: The QC Surrogate Report is the only QC Report that can be generated by an individual TestCode.

14.6.22 **In-House Report** will preview the report selected. **Preview Report** will preview the report selected. **Generate Report** will print the selected reports. **Close** will close the form.

14.7 System Administration

Options for the category are as follows:

14.7.1 Change Access Password – This allows the user to change the password used to login to LIMS.

14.7.2 Clean Tmp Tables – This cleans the temporary tables that the LIMS utilizes in its functions.

14.7.3 LIMS Setup – This is used to configure the workorder prefix, the workorder identification length, and the sample identification length. This is done initially at the time LIMS is setup. This should not be changed.

14.7.4 Remake TmpDataEntry Tables – This remakes the temporary tables used for data entry. This should only be used by a system administrator.

14.7.5 Set (Reset) Invoice Number – This sets (or resets) the starting invoice number. This option is not currently being used.

14.7.6 Set (Reset) Vendor PO Number – This sets (or resets) the vendor purchase order number. This option is not currently being used.

14.7.7 System CalcCheck – This allows a system check of the calculations used by LIMS.

14.7.8 View System Queries – This option allows the user to view the system queries that the LIMS utilizes. This is password secured, and can only be viewed by a system administrator.

14.8 LIMS Configuration

Options for the category are as follows:

14.8.1 Analyte Types – A listing of the analyte types and description of each type. This is a data table, and should only be modified by an administrator.

14.8.2 Analytical reporting Units – A listing of the reporting units that used by LIMS. This is a data table, and should only be modified by an administrator.

14.8.3 Bottles – A listing of the various bottle types, description of the type, uses for the bottles, and the matrix for each type. This is a data table, and should only be modified by an administrator.

14.8.4 Control (Ctrl) Keys – A listing of keyboard shortcuts that allows the user to easily navigate through LIMS. For example, Ctrl-W will open the Workorder form (Sect. 14.4).

14.8.5 Coolers – A listing of laboratory coolers and description for tracking purposes. This is not currently being used.

14.8.6 Data Report Options – A listing of the analytical data reports currently available (Sect. 14.6). This is a data table, and should only be modified by an administrator.

14.8.7 Departments – A listing of the individual departments/description within the laboratory. This is a data table and should only be modified by an administrator.

14.8.8 Documents – A listing of the standardized documents that could be used by LIMS (see Attachment 8). These documents can be added/modified as necessary. Select a document type from the drop down list (Cover Letter, Invoice, Narrative, or Subcontract) to view those document types. Click on the **Add** button on the toolbar to add a document.

14.8.9 Instruments – A listing of the instruments used in the laboratory (see Attachment 9).

14.8.9.1 Click on the **Add** button on the toolbar to add an instrument. Type in the name of the instrument, select an instrument type from the drop down list, and fill in all relevant information.

14.8.9.2 The "Maintenance Log" tab allows analysts to enter and track all instrument maintenance performed. This is currently not being used.

14.8.9.3 The "IDL Limits" tab allows analysts to enter Instrument Detection Limits. This is currently not being used.

14.8.9.4 The **Maintenance Log** button will preview a report containing all maintenance performed and entered into LIMS. This is currently not being used.

14.8.9.5 The **Corrective Action** button links to the corrective action form. See Sect. 14.14.2 for more information.

14.8.10 Load / Upload MDLs – A mechanism to load MDL or PQL data into LIMS from an Excel spreadsheet for a given test code. This is currently not being used.

14.8.11 Matrices – A listing of the various matrices used by LIMS. This is a data table, and should only be modified by an administrator.

14.8.12 Organization – The laboratory name, mailing address, contact information, phone number, fax number, invoice address, and invoice phone number are entered/modified.

14.8.13 QC Levels – A listing of the different QC Levels used by LIMS. This is a data table, and should only be modified by an administrator.

14.8.14 Sample Storage Areas – A listing of the different storage areas and description for the laboratory. This is a data table, and should only be modified by an administrator.

14.8.15 Sample Types – A listing of the different sample types along with description used by LIMS. This is a data table, and should only be modified by an administrator.

14.9 Laboratory Management

Options for the category are as follows:

14.9.1. Clients – This contains client information (see Attachment 10). Note: There can be no duplicates in the Client ID field.

14.9.1.1 The "Main" tab contains general client information (Company name, address, phone/fax numbers, contacts, etc.).

14.9.1.2 The "Invoice" tab contains client billing information.

14.9.1.3 The "ReportOptions" tab contains client reporting preferences.

14.9.1.4 To review client information, click on the appropriate letter to select all clients with that letter. Select the appropriate client from the list on the left.

14.9.1.5 To add a client, click on the **Add** button on the toolbar. Fill in the Client ID field with a unique client ID. Fill in all relevant information (Sects. 14.9.1.1 – 14.9.1.3).

14.9.2. Invoiced To Date – Allows management to view the amount received or invoiced in a given period. Select a start date and an end date. Click **Received Report** to view spreadsheet containing information on the amount received in that period of time. Click **Invoice Report** to view the amount invoiced in that period. Click **Close** to close the form.

14.9.3. Monthly Client Billing – Allows clients to be billed on a monthly basis instead of a workorder basis. This is currently not being used.

14.9.4. On Time Performance – Allows a quick view of on time performance for the laboratory. Click **View Graph** to view a graphical representation of on time performance. Check or uncheck the Use box to limit the records viewed.

14.9.5. Organization – General laboratory information is entered here. (Same as Sect. 14.8.12)

14.9.6. Project revenue Summary – A project revenue summary report is previewed on the screen. Select a specific project or a wildcard (*) to view all projects.

14.9.7. Projects – Allows projects to be added and reviewed (See Attachment 11). Projects are a useful tool for facilitating project management functions concerning a client specific project.

14.9.7.1 The "Main" tab contains general information regarding the project (client, project name, reporting, QC level, etc).

14.9.7.2 The "Report Info" tab contains company reporting and invoicing information.

14.9.7.3 The "Test Info" tab contains test group (Sect. 14.12.2), test (Sect. 14.12.3), and pricing information relating to the project.

14.9.7.4 To add a project, click the **Add** button on the toolbar, and fill in the appropriate information (Sects. 14.9.7.1 – 14.9.7.3).

14.9.7.5 To review a project, select the client from the drop down list on the left. This brings up all projects for that client. Then select the individual project for review.

14.9.8. Quotes – Allows project management to add or review quotations for analytical services (see Attachment 12).

14.9.8.1 The "Main" tab contains general information regarding the client, project and laboratory services needed.

14.9.8.2 The "Tests" tab contains information regarding the analytical test to perform, and the test pricing.

14.9.8.3 The **Copy Quote** button allows a previous similar quote to be copied. Select the quote to copy.

14.9.8.4 The **Bottles** button links directly to the bottle order form (see Sect 14.10.1).

14.9.8.5 The **Print** button generates a hardcopy of the Quotation for Analytical Services report.

14.9.8.6 Quotations can be quickly sorted by their status. Click the **Lost** button to view all quotes that were lost. Click the **Pend** button to view all quotes that are pending. Click the **Won** button to view all quotes that were won. Click the **All** button to view all quotes.

14.9.9. Revenue By CLIENT – Runs a query that generates revenue by client, listing the largest customers first.

14.9.10. Revenue By PROJECT – Runs a query that generates revenue by project, listing the largest projects first.

14.9.11. Work In Progress (WIP) Report – This generates a summary report for all in-house work. Workorders are ordered by due date, listing the oldest ones first. The individual laboratory departments are listed across the top. The number of outstanding tests shows up as a number under each department for each workorder listed.

14.9.12. Work Order - Status – Gives a status of workorders (see Attachment 13).

14.9.12.1 Click **LoginReview** to view all workorders waiting to be login reviewed.

14.9.12.2 Click **Completed** to view all workorders that have been login reviewed but are not completed.

14.9.12.3 Click **Validated** to view all workorders that have been login reviewed and completed, but are not validated.

14.9.12.4 Click **Reported** to view all workorders that have been login reviewed, completed, and validated, but are not reported.

14.9.12.5 Click **Invoiced** to view all workorders that have been login reviewed, completed, validated, and reported, but are not invoiced.

14.9.12.6 Click **All** to view all workorders.

14.9.13. Workload History – Generates a workload history report for a specified period. Select the appropriate start and end date. Key in on the date the samples were received or the date the samples were due. Select an appropriate department (or test code). Click **Preview Report** to preview the Workload History Report. Click **Generate Report** to print the Workload History Report. Click **Close** to close the form.

14.10 Coordination

Options for the category are as follows:

14.10.1. Bottle Orders – Track client requested bottle orders. This is currently not being used.

14.10.2. Check Subcontracted Work – Generates a summary of subcontracted work, listing the laboratory sample number, test code, and subcontractor.

14.10.3. Client Reports – See Section 14.6

14.10.4. Create Data Export Specification – Creates a specific data export format. Select the client, project, and workorder as necessary. Select the format of the exported data. Select a unique name to save the specific export format. Click **OK**.

14.10.5. Export Data – Generates an Electronic Data Deliverable. Select a specification (See Section 14.10.4), select workorder or project, and a destination for the output. Click **OK** to generate the deliverable.

14.10.6. Laboratory Backlog Report – See Section 14.2

14.10.7. Monthly Client Billing – See Section 14.9.3

14.10.8. Sample Disposal – Track the disposal of samples from individual workorders. This is currently not being used.

14.10.9. Samples Currently on HOLD – A summary of sample and test that were placed on hold.

14.10.10. SDG Sample Assignment – Create Sample Delivery Group assignments to laboratory samples. This is currently not being used.

14.10.11. View Duplicate Run Data – Mechanism to view duplicate analytical run data for individual samples and test. Select a workorder from the drop down list. Select a sample number and

test code form the list on the left. The duplicate data shows up on the right. This is currently not being used.

14.10.12. View Sample SEL List – Generates a sample selection list report, listing the laboratory sample number with reportable analytes. Select a test code from the drop down list. Select the appropriate laboratory sample number. Click **View SEL** to view a report listing the analytes that need to be reported. Click **Close** to close the form.

14.10.13. Work In Progress (WIP) Report – See Section 14.9.11

14.10.14. Work Order - Status – See Section 14.9.12

14.10.15. Workload History – See Section 14.9.13

14.11 Analytical

Options for the category are as follows:

14.11.1. Chemical Inventory – Allows users to track the chemical reagents used and stored within the laboratory. This function is currently not being used.

14.11.2. Export Samples to Excel – See Section 14.5.12

14.11.3. Import Specifications – See Section 14.5.11

14.11.4. Instruments – See Section 14.8.9

14.11.5. Sample Prep – See Section 14.3

14.11.6. Sample Prep: INCOMPLETE – A query that lists all samples for which preparation has started, but is not yet complete.

14.11.7. Spikes / Standards – Allows users to log and track spike solutions and standard solutions within LIMS. This function is currently not being used.

14.11.8. View Duplicate Run Data – See Section 14.10.11

14.11.9. View Sample SEL List – See Section 14.10.12

14.11.10. Data File Formatter – A program allowing certain instrument output files to be read and re-formatted into a file that can be directly imported into LIMS. Select an instrument from the drop down list, select the file to format, select the Test Code, and click the format button. The program will stop and prompt the user to select a Test Code for each sample. After reading the samples, the program will save the original output file with a new name and save the newly formatted file for importing. Click  to exit the program. Note: This program is not available on all the computers in the laboratory.

14.12 Test Information

Options for the category are as follows:

14.12.1. Analyte Table – A listing of the reportable Test Numbers, analyte types, analytes, and other information. This is a data table, and should only be modified by an administrator.

14.12.2. Test Groups – Test Groups are groupings of individual tests (See Attachment 14 and Section14.12.3). For example, a TCLP analysis would require one test code for TCLP tumbling, another test code for digestion/extraction, and another test code for analysis. Grouping tests together helps facilitate the log-in process, and other project management functions.

14.12.2.1To review a test group, first select either **1. Act** (active) or **2. InAct** (inactive).

14.12.2.2Select the appropriate test group from the list displayed under the index. A listing with the Test Group, Group Name, and individual Test Codes for that group will be displayed.

14.12.2.3To add a test group, click the **Add** button on the toolbar.

14.12.2.4Type in a unique name for the Test Group. Fill in the Group Name, matrix, bottle, and comments as appropriate.

14.12.2.5 Select Test Codes from the drop down list by clicking on the right side of the Test Code field.

14.12.2.6To select specific analytes or compounds to report, click the SEL box. Change the Report column to Y (reported) or N (not reported). This only works with analysis Test Codes.

14.12.2.7 Change the QuotedPrice as needed.

14.12.2.8Repeat steps 14.12.2.5 through 14.12.2.7 for any additional Test Codes.

14.12.2.9Click on **ReCALC** to recalculate the price.

14.12.2.10 Click on **Copy Group** to copy an existing group into a new Test Group. Select the Test Group to copy from and type in the new Test Group name. Click **Create TestGroup** to create the Test Group.

14.12.3. Tests – Test Codes is where information concerning preparation and analysis is stored within LIMS. See Attachment 15.

14.12.3.1Click Select All  to select all the Test Codes.

14.12.3.2Select either **1. Act** (active) or **2. InAct** (inactive).

14.12.3.3To filter the Test Codes by department, select a department from the drop down list.

14.12.3.3Select a Test Code from the index on the left.

14.12.3.4The "Main" page of the Test Code contains information on the reference, department, holding time (from collection, from receipt, and from analytical preparation), sample bottle, reportable test number, matrix, pricing (list price and individual analyte price), and reportable units. The information on the bottom of the main page will change depending on whether the Test Code is a preparation Test Code or an analysis Test Code.

14.12.3.4.1The first form in attachment 15 displays the information for a preparation Test Code. Linked analytical Test Codes, and sample preparation amounts are entered here.

14.12.3.4.2The second form in attachment 15 displays analytical Test Code information. This displays information from the Analyte table noted in 14.12.1.

14.12.3.5The "Limits" page of the Test Code contains information on the analyte type, MDL, RL (PQL), and Upper Quantitation Limit (UQL).

14.12.3.5.1Click **Load** to load the analytes listed on the "Main" page.

14.12.3.5.2Click **Delete** to delete the current list of analytes from the Test Code.

14.12.3.5.3Click **Copy** to copy analytes and limits from an existing Test Code. Select a Test Code to copy from the drop down list that appears next to the **Copy** button.

14.12.3.5.4Click **Del Extras** to remove certain analytes. This is currently not being used.

14.12.3.5.5Click **Update** to load and update MDL information (See Sect.14.8.10). This is currently not being used.

14.12.3.5.6Click **UpdateSEL** to update select list, MDL, PQL, and UQL information to this Test Code as it is stored in Test Groups, Projects, Quotes, and currently logged in samples. This is currently not being used.

14.12.3.5.7Click **Print** to print a Method Detection/Reporting Limits report for the Test Code.

14.12.3.6The "Specs" page of the Test Code contains information on analyte and surrogate spike levels, recovery, and RPD information for different sample types. Select an existing Sample Type on the left, and view/edit the information for that Sample Type on the right.

14.12.3.7 The "Results / CLP" page contains information on how the Test Code will be reported. The CLP Reporting Parameter section is currently not being used.

14.13 Sales

Options for the category are as follows:

14.13.1. Bottle Orders – See Section 14.10.1

14.13.2. Clients – See section 14.9.1

14.13.3. Projects – See Section 14.9.7

14.13.4. Quotes – See Section 14.9.8

14.14 Quality Control

Options for the category are as follows:

14.14.1. Control Charting – Allows for Quality Control Charting (See Attachment 16), and automatic update of limits in LIMS.

14.14.1.1 The "RETRIEVE DATA" page allows the user to filter and select the Test Codes to query.

14.14.1.1.1 Select a department from the drop down list.

14.14.1.1.2 Select a matrix from the drop down list. The available Test Codes will display on the left.

14.14.1.1.3 Select the number of points for LIMS to retrieve.

14.14.1.1.4 Highlight the Test Codes to query by clicking on the Test Code.

14.14.1.1.5 Click the box under the Use column to select the Sample Types. Clicking the CAL radio button allows the user to select instrumental Sample Types.

14.14.1.1.6 Click **Get Data** for LIMS to retrieve the data for the options chosen.

14.14.1.2 The "VIEW DATASET / GRAPH" page displays the data retrieved for the selections in Section 14.14.1.1.

14.14.1.2.1 Select a graph type: **REC** is recovery, **RPD** is relative percent difference.

14.14.1.2.2 Click **Spikes**, **Surrogate**, or **All**.

14.14.1.2.3 Highlight an analyte by clicking on the appropriate analyte. The data for that analyte will be displayed on the right. Right click the analyte to display the graph. The statistics for that analyte are displayed under the analyte index.

14.14.1.2.3 Click **Grubbs Outlier** to statistically evaluate the dataset.

14.14.1.3 The "PLOT GRAPH / UPDATE LIMITS" allows for printing reports and automatically updating limits within LIMS. The data will be displayed on the right with current limits under the Old columns and newly calculated limits under the New columns.

14.14.1.3.1 Select an analyte to view or update by checking the box in the UP column of the dataset.

14.14.1.3.2 Click **PLOT RPD Graph(s)** to print an RPD Quality Control Report listing the data and graphs.

14.14.1.3.3 Click **PLOT REC Graph(s)** to print a REC Quality Control Report listing the data and graphs.

14.14.1.3.4 Click **Update TESTS** to automatically update the Test Code(s). This will update the Test Code and Sample Types that have been selected on the left (should be the same selections that chosen in Section 14.14.1.1).

14.14.2. Corrective Action Reports – The process for generating a Corrective Action Report (CAR) is described in STAT SOP 230 Corrective Actions (See Attachment 17).

14.14.2.1 Click **Add** on the toolbar to add a new record.

14.14.2.2 Select a Department, Instrument ID, Analytical Run ID, and Batch ID from the drop down lists as appropriate.

14.14.2.3 Type in the CAR summary. Note: This field is limited to 75 characters.

14.14.2.4 Select the individual initiating the CAR, and the date initiated.

14.14.2.5 Fill in the Complete Description of Nonconformance, and Corrective Action Required sections.

14.14.2.6 Fill in other sections as the CAR is completed and reviewed.

14.14.2.7 Click **Copy to Narrative** to update appropriate case narratives.

14.14.2.8 Click **Print Report** to preview the Corrective Action Report.

14.14.3. QA Validation – This runs a query to determine all samples and tests that have been completed by the analyst, and are awaiting a secondary review for validation. Double click the Work Order next to the Test Code to view the analytical run.

14.14.4. QC Report – See section 14.6

14.15 Reporting

Options for the category are as follows:

14.15.1. Batch Reporting – Allows for multiple WorkOrders to be reported and invoiced. This is currently not being used.

14.15.2. Client Reports – See Section 14.6

14.15.3. Create Data Export Specification – See Section 14.10.4

14.15.4. Export Data – See section 14.10.5

14.15.5. QC Report – See Section 14.6

14.15.6. Work In Progress (WIP) Report – See section 14.9.11

14.16 Operations

Options for the category are as follows:

14.16.1. Bottle Orders – See Section 14.10.1

14.16.2. Chemical Inventory – See Section 14.11.1

14.16.3. Cooler Tracking – Allows for tracking of coolers the laboratory has shipped out. This is currently not being used.

14.16.4. Instruments – See Section 14.8.9

14.16.5. Vendor Purchase Orders – Allows for automatic generation of Purchase Orders for supplies. This is currently not being used.

14.16.6. Vendors – A listing of laboratory suppliers/supplies. This is currently being used as a reference for purchasing.

14.17 Changing a Record in LIMS

The procedure to change a record follows. With the exception of Data Entry, all locked records will display with  . Data Entry records are locked when a check mark is listed under the QA column. Note: you must be logged on with your account and have the permission level to change a record.

14.17.1 Unlock the record by pressing  , and enter your password. Under Data Entry, click **QA Auth** and enter your password.

14.17.2 Change the record as necessary. LIMS automatically tracks the date modified and the user making the change.

14.17.3 If data has been modified in a record under Data Entry, LIMS automatically tracks the before and after values in a separate table, as well as the individual making the change.

14.18 Administrator Duties and Responsibilities

14.18.1 Account Management / Access – The Technical Manager is responsible for adding/removing users from LIMS. The new user is added first to the LabPersonnel table. They are then added to a special file used to access LIMS. See Section 14.18.4.

14.18.2 Routine Maintenance

14.18.2.1 Weekly – Each individual workstation running the LIMS should be compacted. Go to the Main Menu screen. On the menu across the top, click *Tools*, *Database Utilities*, and *Compact Database*. The need to perform this operation is dependent on the usage of the LIMS at the individual workstation.

14.18.2.2 Monthly – The main database stored on the server should be compacted per 14.18.2.1.

14.18.2.3 Annually – The main database stored on the server will be reviewed for content. Employees leaving STAT Analysis will be rendered inactive, and their access to LIMS will be removed.

14.18.2.4 As needed – The main database will be reviewed for space, and records will be archived. Occasionally, the main database will become corrupted. The database can normally be repaired by clicking *Tools*, *Database Utilities*, and *Repair Database*, or the database may be restored from a tape backup (See Sect. 14.18.8).

14.18.3 Changes / Upgrades – Changes or upgrades to LIMS are performed as needed. The Technical Manager, in conjunction with the Vendor, will determine what changes are to be made to the working database. Two copies of the working database exist: The Technical Manager maintains a copy, and a copy is stored on the backup server. LIMS changes will be made to the Technical Manager's copy and tested on the situation that necessitated the change. If this test is successful, the change will be incorporated into LIMS. The server copy is updated only upon completion of a successful test.

14.18.4 Security – Security for LIMS is two-fold.

14.18.4.1 Microsoft Access® provides inherent security within a special file. This file is used to initially access LIMS. Users are added based on their last name (or first initial and last name) and are assigned to User Groups depending on their responsibilities and functions. A password is required.

14.18.4.1 LIMS provides for security within the database by requiring a second password to access certain functions. These departments are assigned by the Technical Manager in conjunction with the Laboratory Director. For example, a person with QA Authority will not be able to change a Workorder (Project Management authority).

14.18.5 Communication with Vendor / Vendor Support – STAT Analysis currently does not maintain a service agreement with the Vendor. The Vendor has a copy of the STAT Analysis database, and can offer technical phone support.

Vendor Contact: Khemia Company
Doyce T. Blair
8247 W. 99th Ave
Broomfield, CO 80021
TEL: (303) 438-1448
Email Address: DTBlair@Khemia.com

14.18.6 Training – Training on this SOP will be conducted according to STAT SOP 1230 Training.

14.18.6.1 New Employee – New employees should be trained on this SOP within one month of hire date.

14.18.6.2 Retraining – A major revision to this SOP requires retraining.

14.18.7 Information Audit – The QA Manager, as part of the Internal QA Audit process, reviews 5% of the data produced in the laboratory. Part of this review encompasses the information entered into LIMS. When deficiencies are noted by the QA Manager during his internal audit, the corporation's corrective action process is employed to remedy these situations.

14.18.8 Information Back-up – The main database is backed up daily onto tape, Monday – Friday. See STAT SOP 1500 Computer Network.

14.18.9 Information Archive – Archiving of the data in LIMS depends on the workload history and the usage of the database (currently about one year of data is stored in the active database). The archiving is done on an as needed basis. Data from the current database is transferred into an archive database, and then removed from the current database. The archived database is stored on the server for access. All users can access and review the data in an archived database, however only users with administrator rights can modify the data.

15. Data Reduction, Calculations and Loading

Not Applicable to the SOP

16. Method Performance

Not Applicable to the SOP

17. Pollution Prevention

Not Applicable to the SOP

18. Data Assessment and Criteria for Quality Control Measures

Not Applicable to the SOP

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the Data Review findings determine that the laboratory performance for a particular parameter is judged to be out of control, the problem must be immediately identified and corrected. The analytical results produced for that parameter are suspect. The time frame for the out of control situation will be determined. This will identify what client samples were analyzed during the time frame. Immediate corrective action includes written notification to clients that the data produced for the parameter may be affected and, if possible, to the degree that the data was affected.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data are revealed through the Data Review process, the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discuss options with the client. Reanalysis or reporting the data with qualification are alternatives. This may include the re-issue of amended reports with qualified data indicating that the previously reported results did not meeting the laboratory defined criteria. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting. For amended reports, the reason for the report amendment is clearly identified and explained.

Final data results must be qualified in the client report for results not meeting the laboratory-defined criteria.

The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and recorded by the Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21. Waste Management

Not Applicable to the SOP

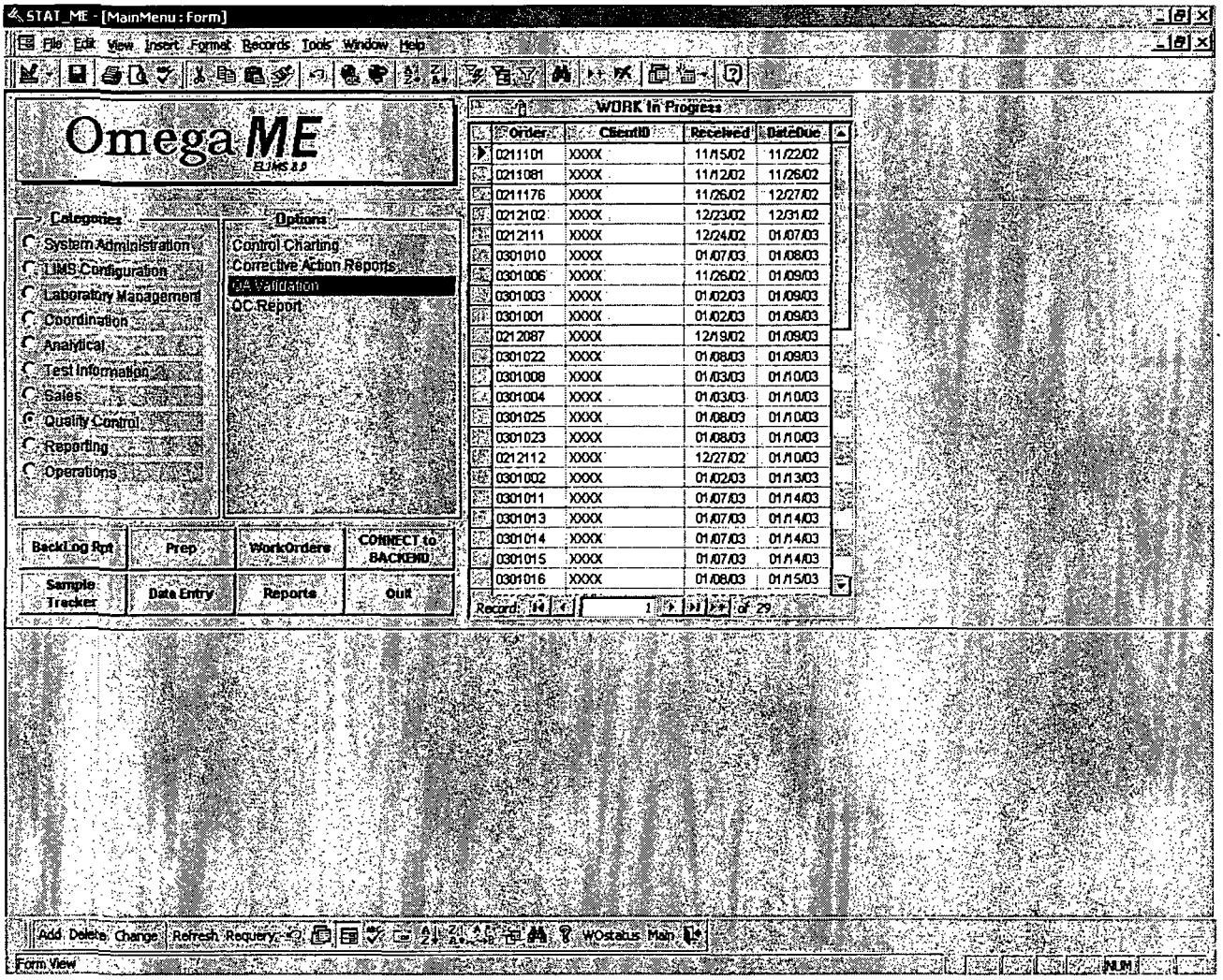
22. References

22.1 SOP 230 Corrective Actions
22.2 SOP 300 Sample Receiving
22.3 STAT SOP 1220 Internal Quality Assurance Audit
22.4 STAT SOP 1500 Computer Network
22.5 STAT Analysis Corporation Quality Assurance Manual
22.6 STAT SOP 1230 Training

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

Attachments are as follows:

ATTACHMENT 1:



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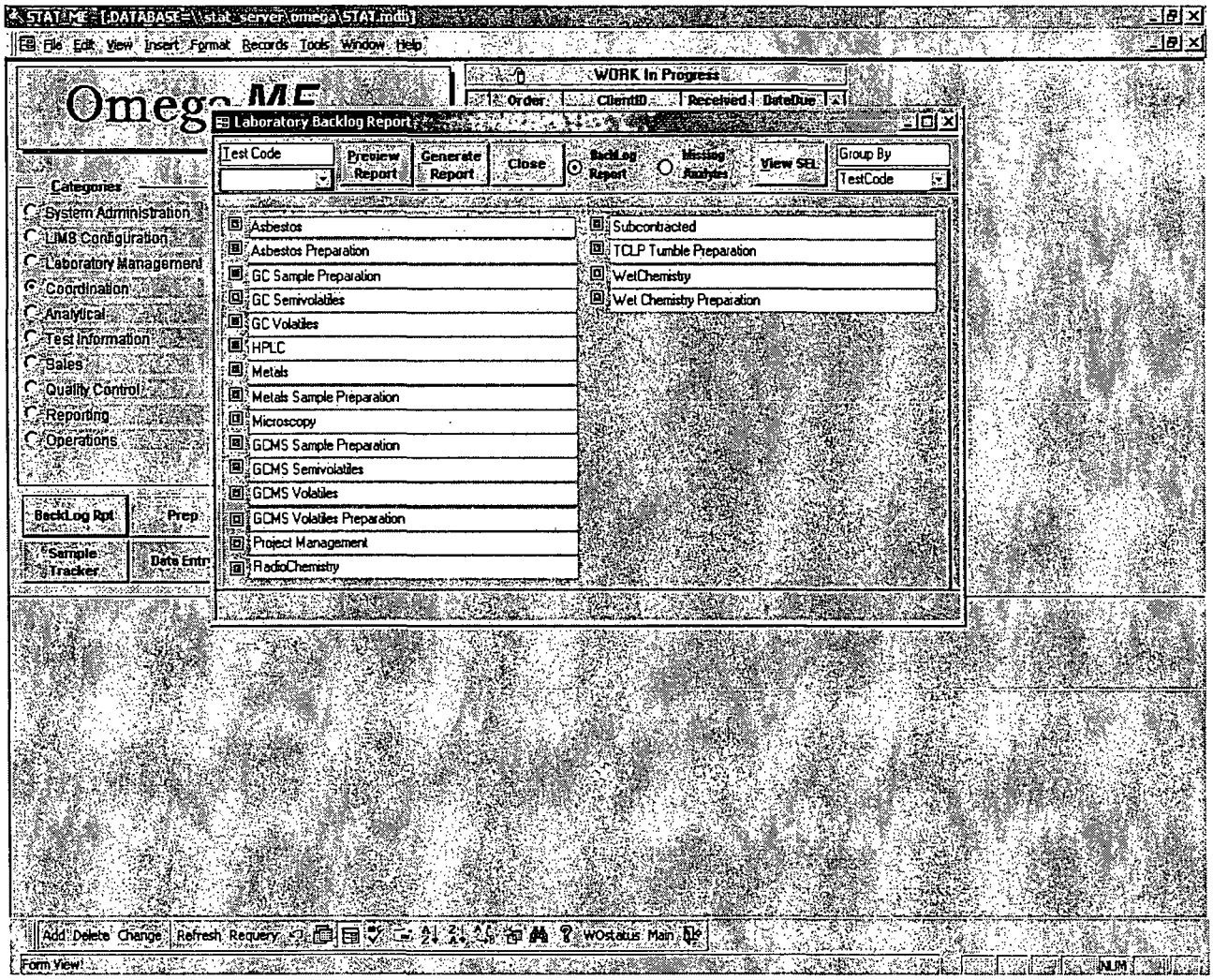

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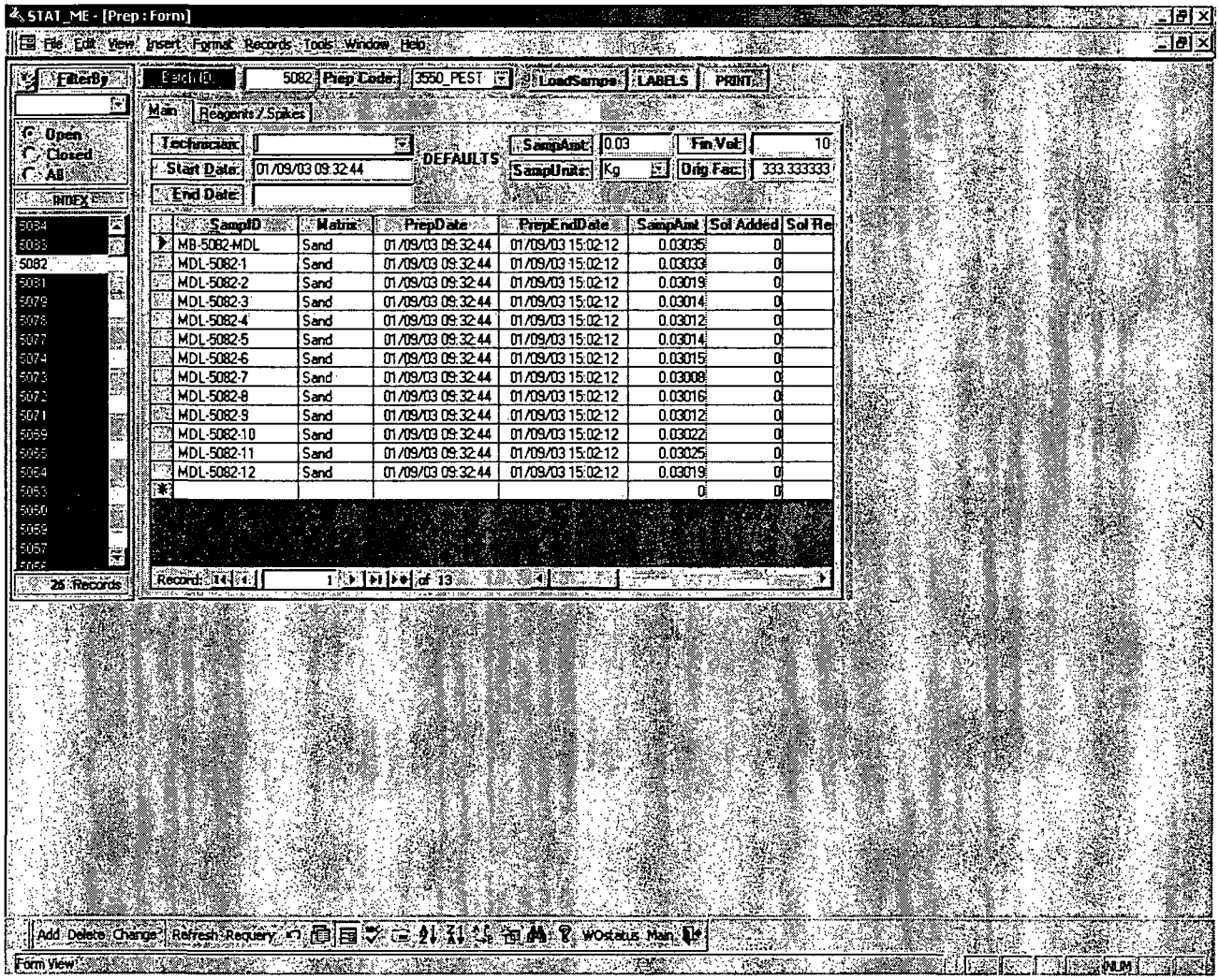

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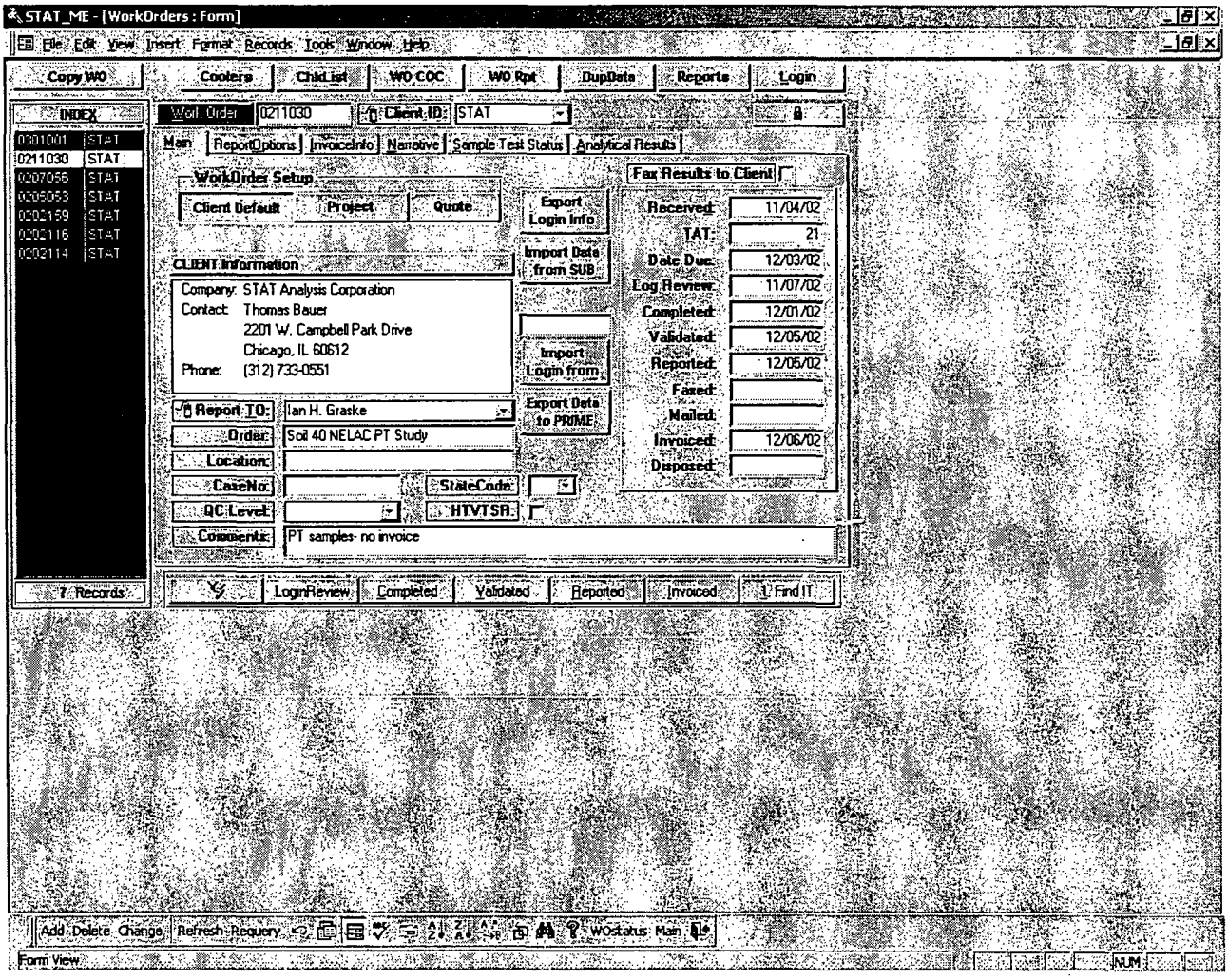

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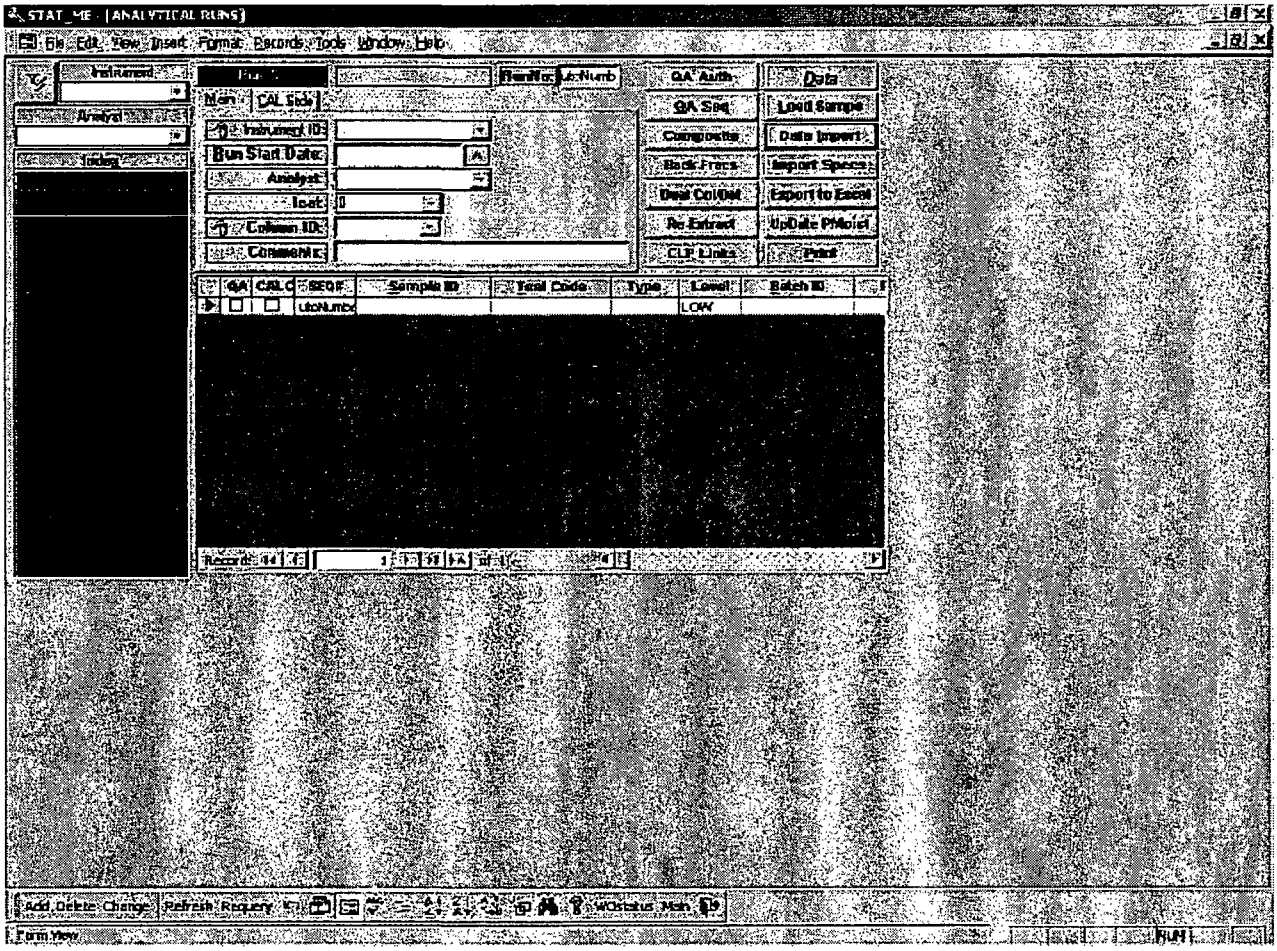

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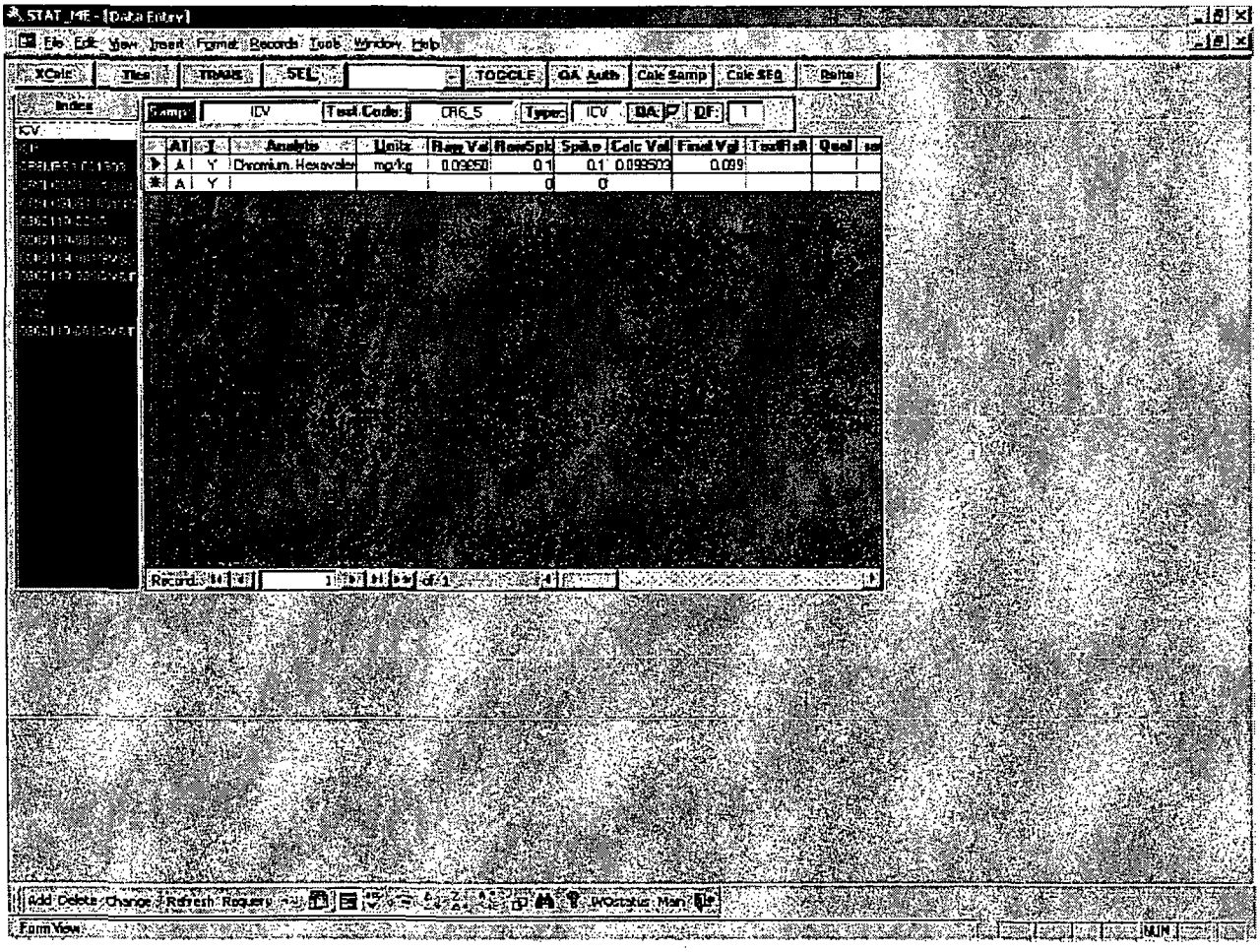

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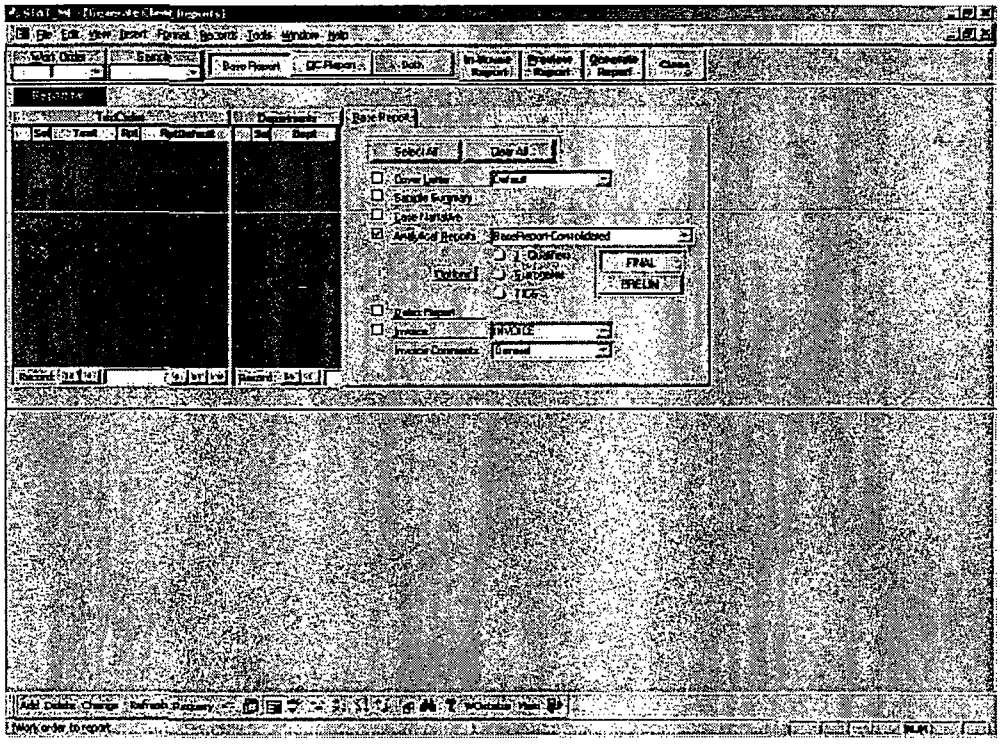

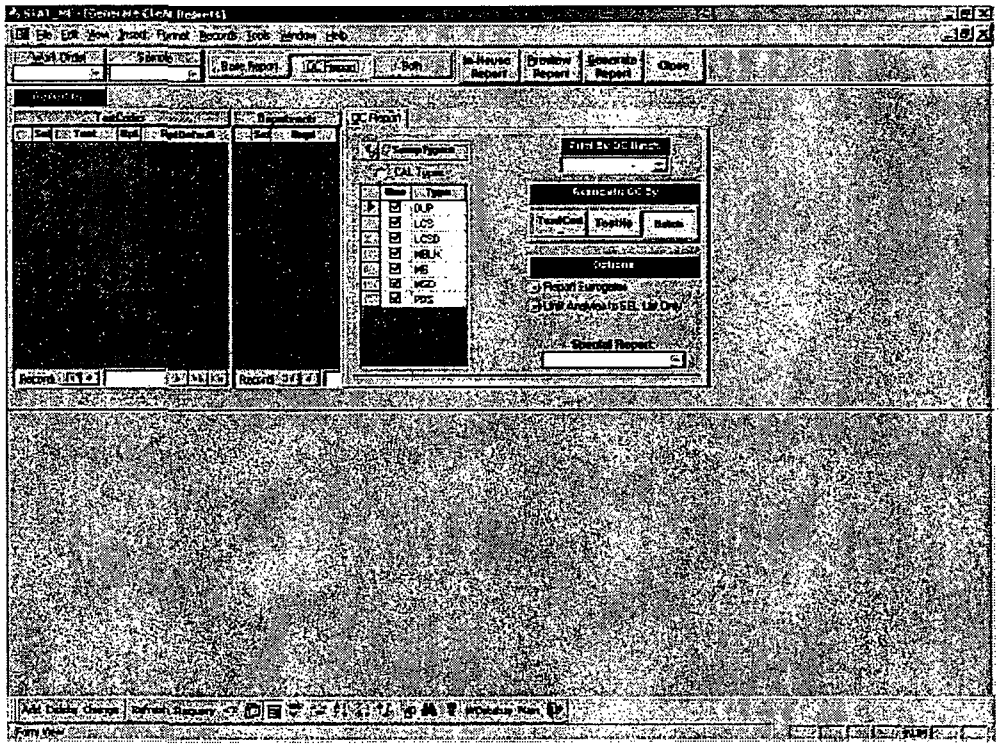

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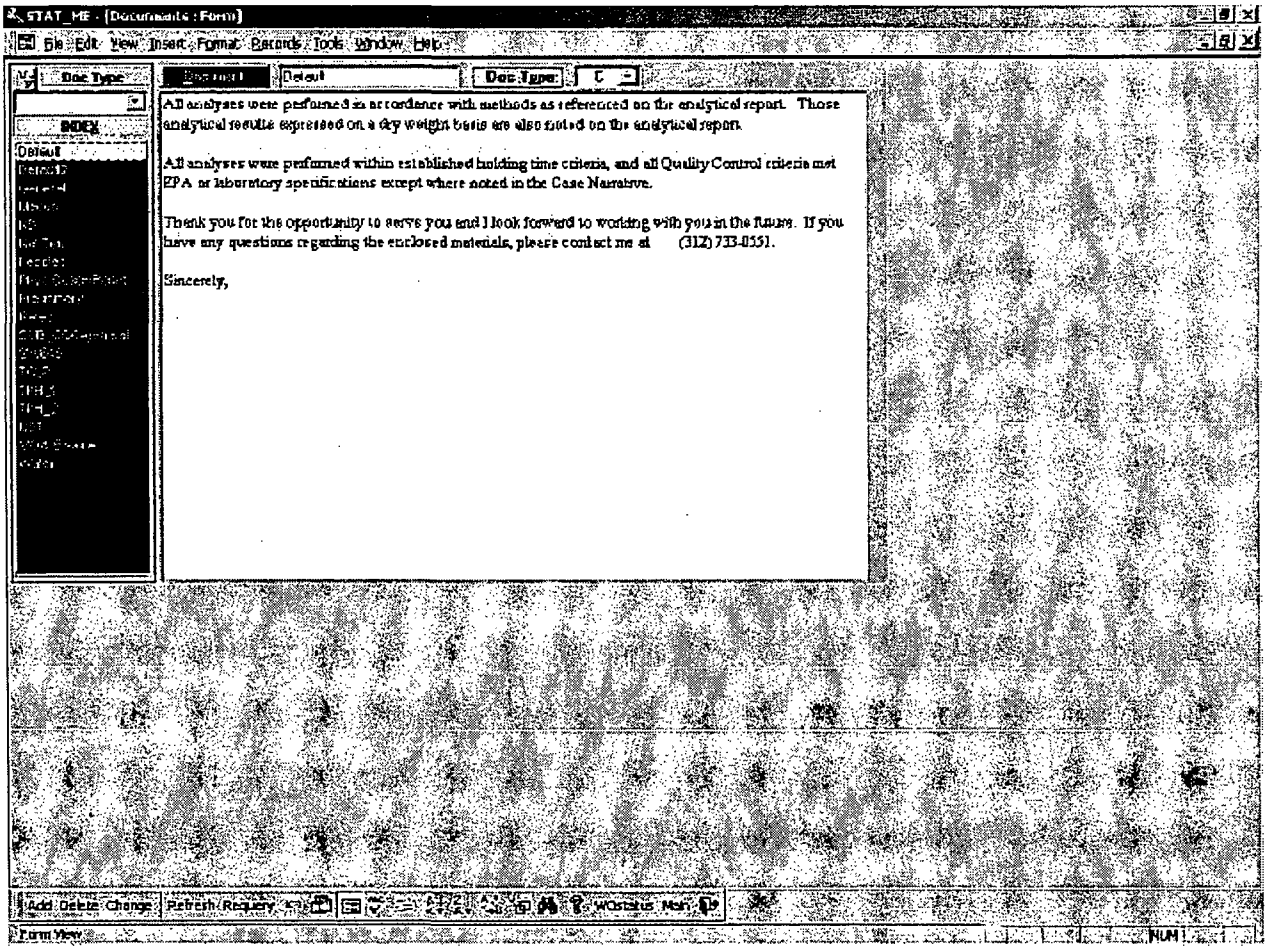

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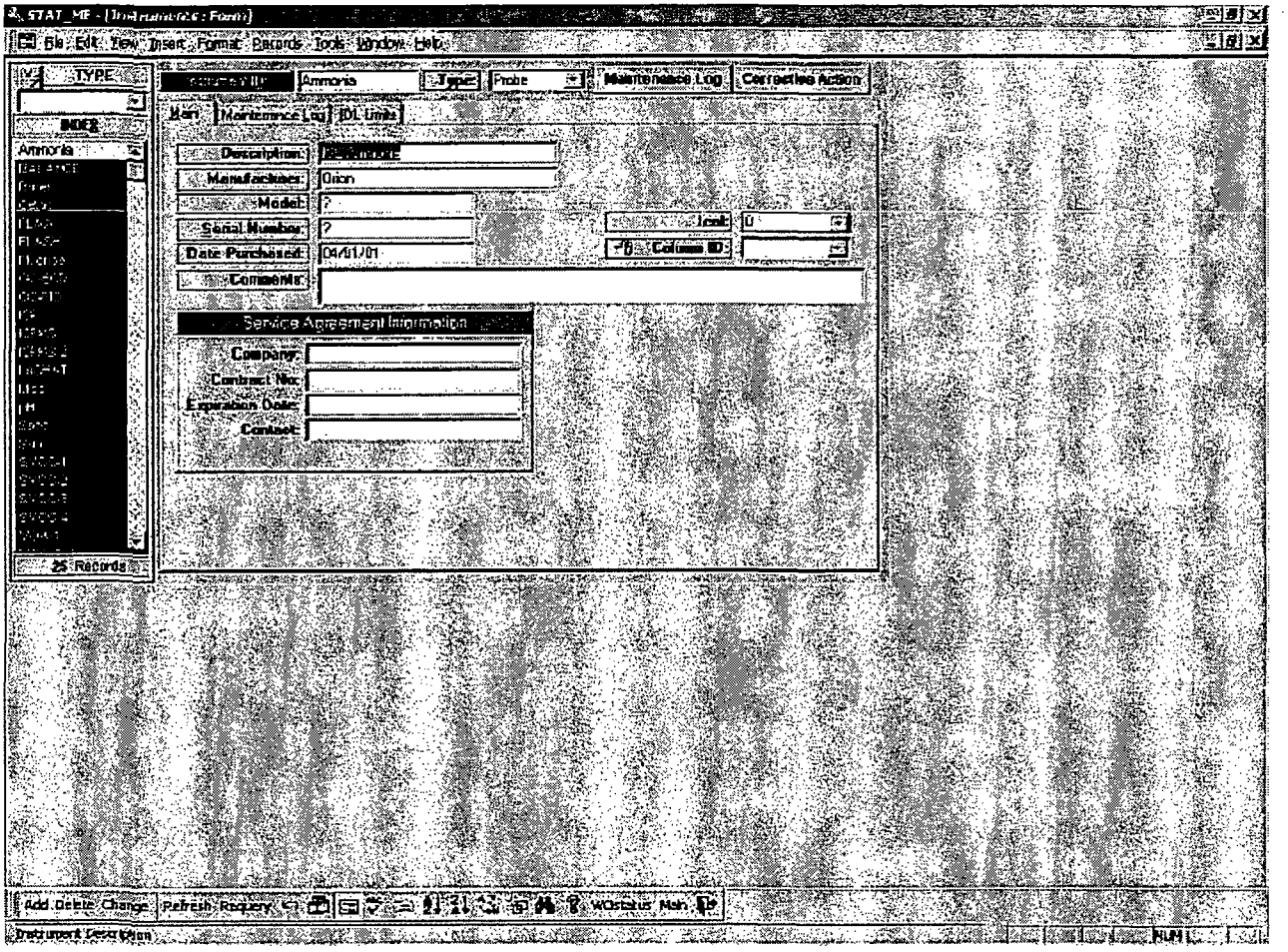

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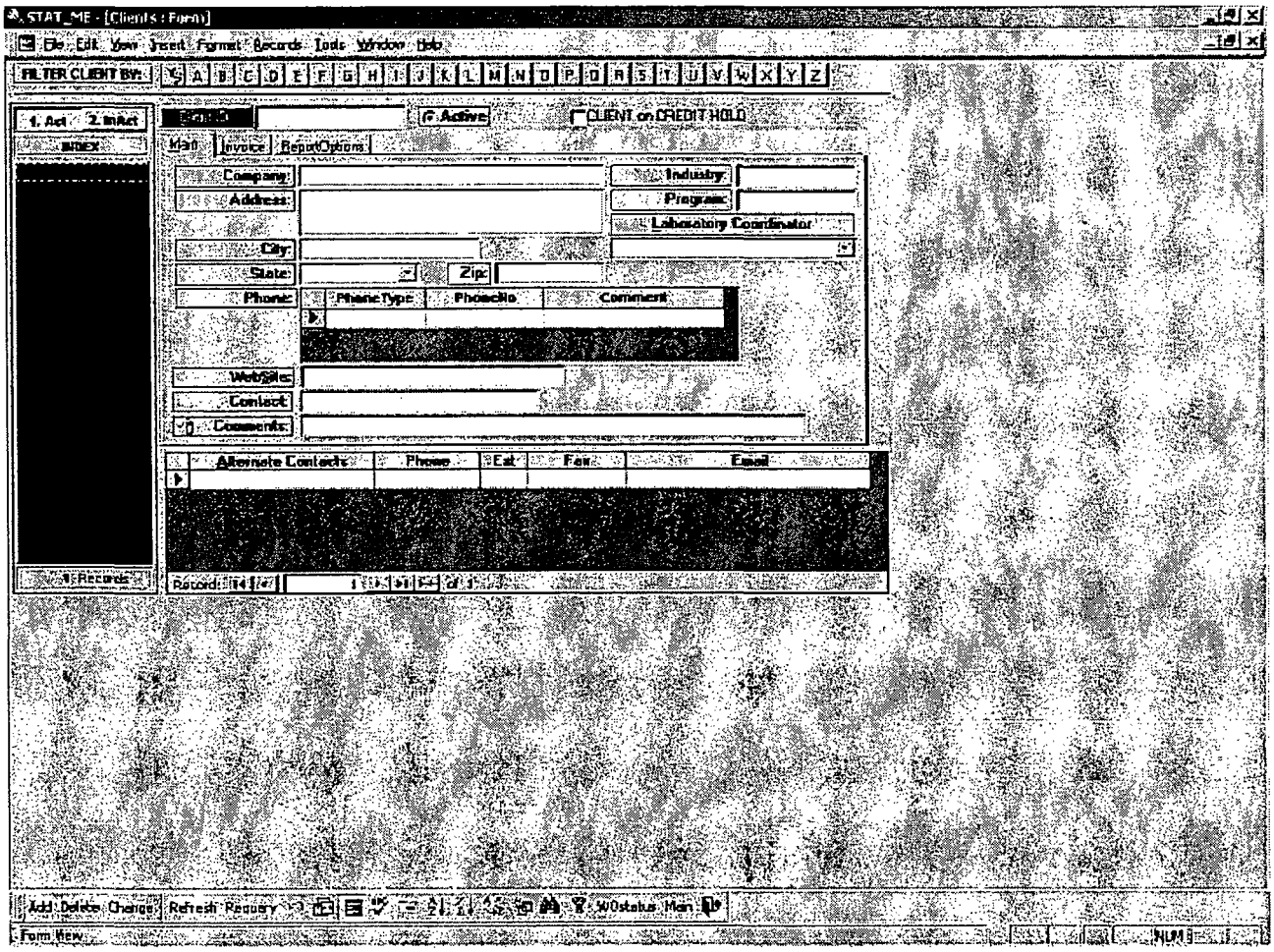

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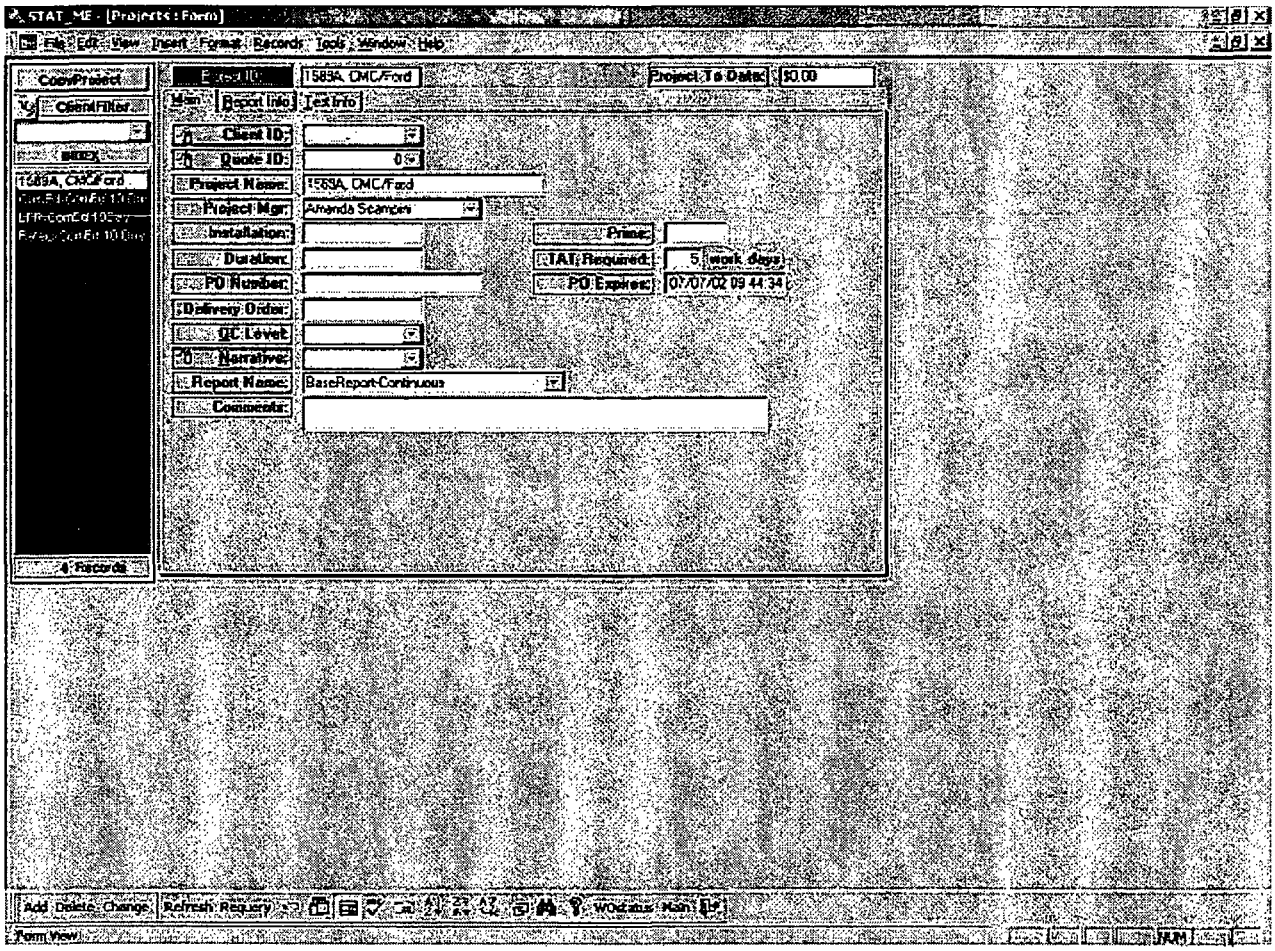

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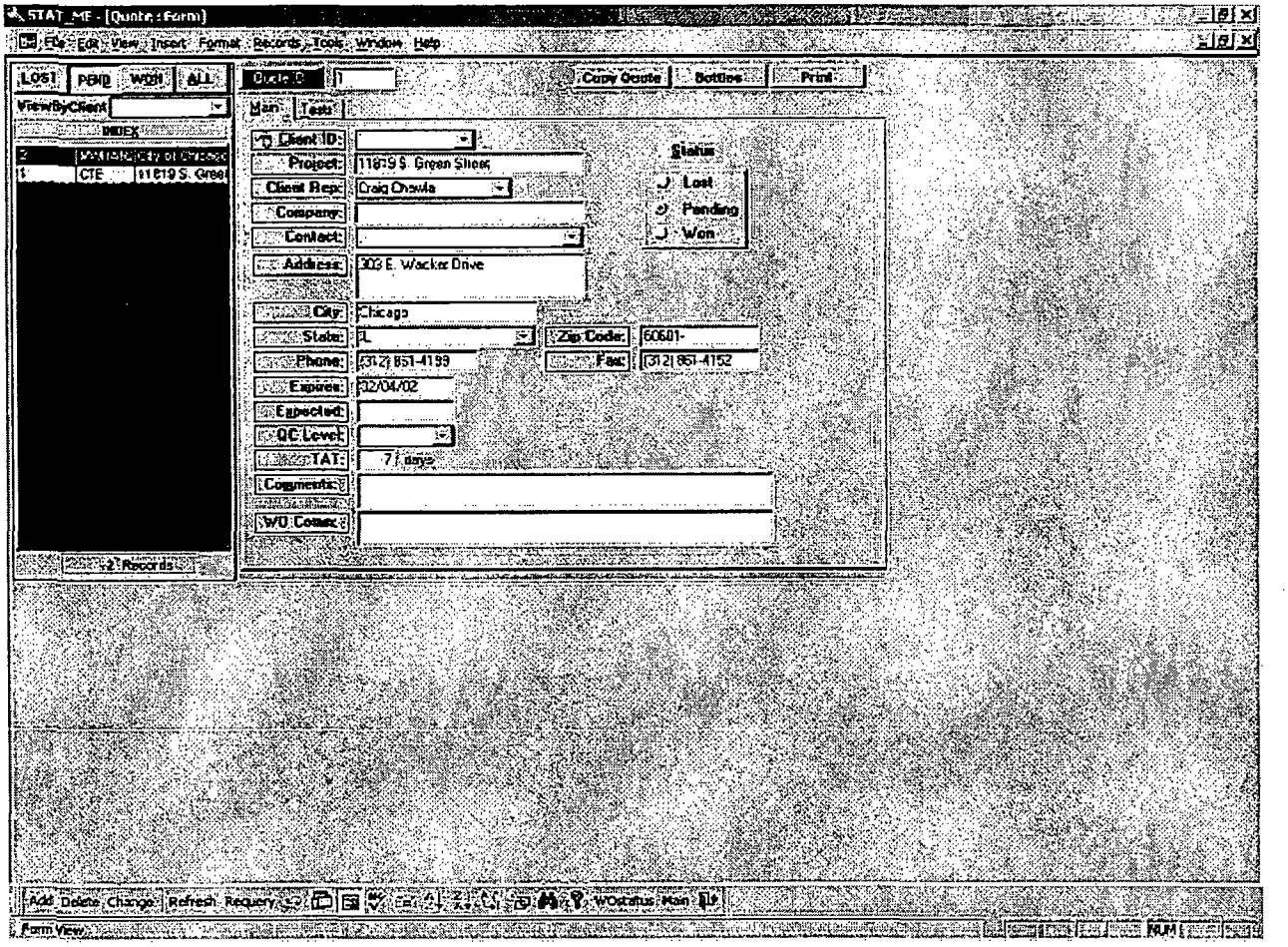

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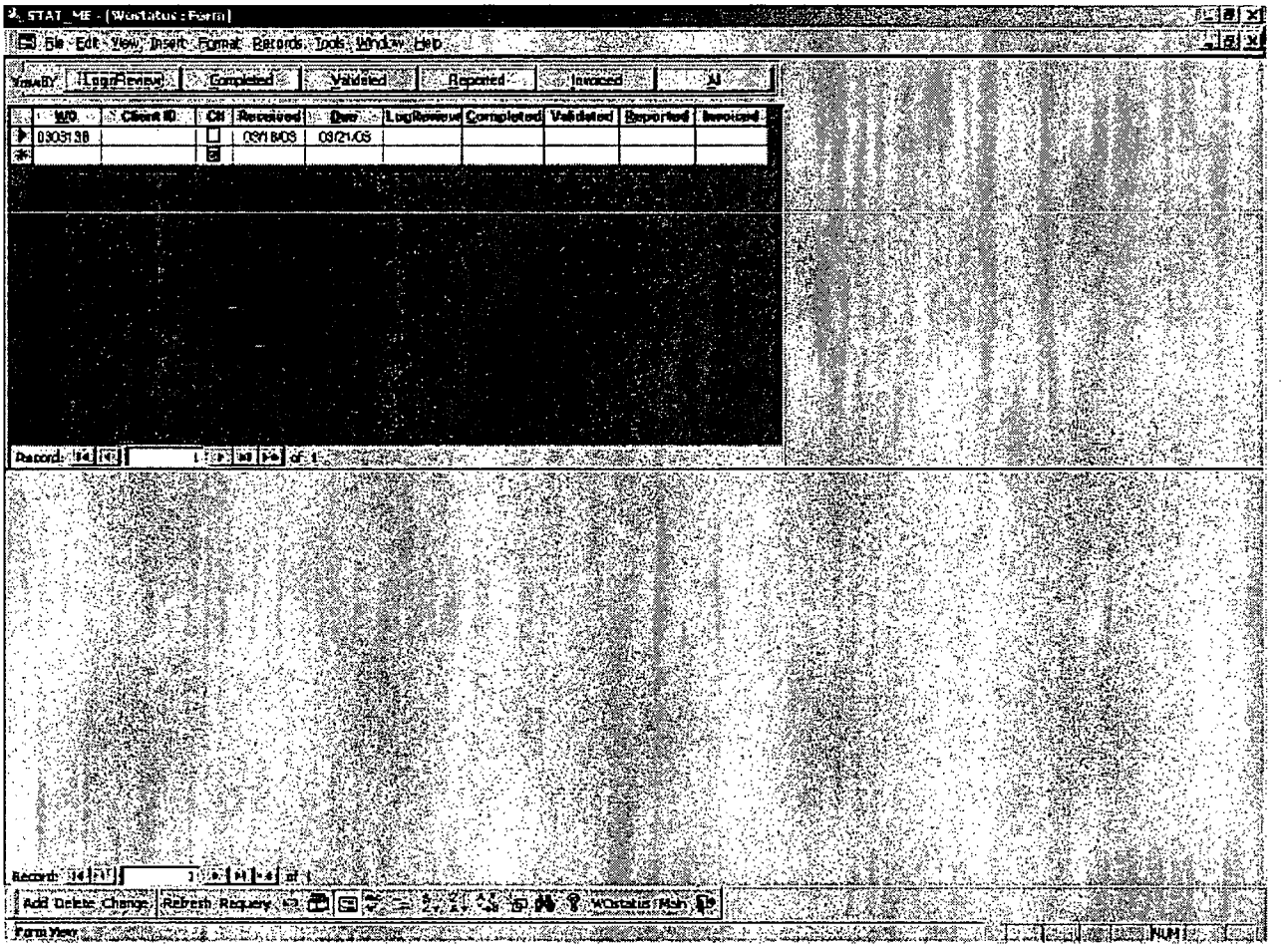

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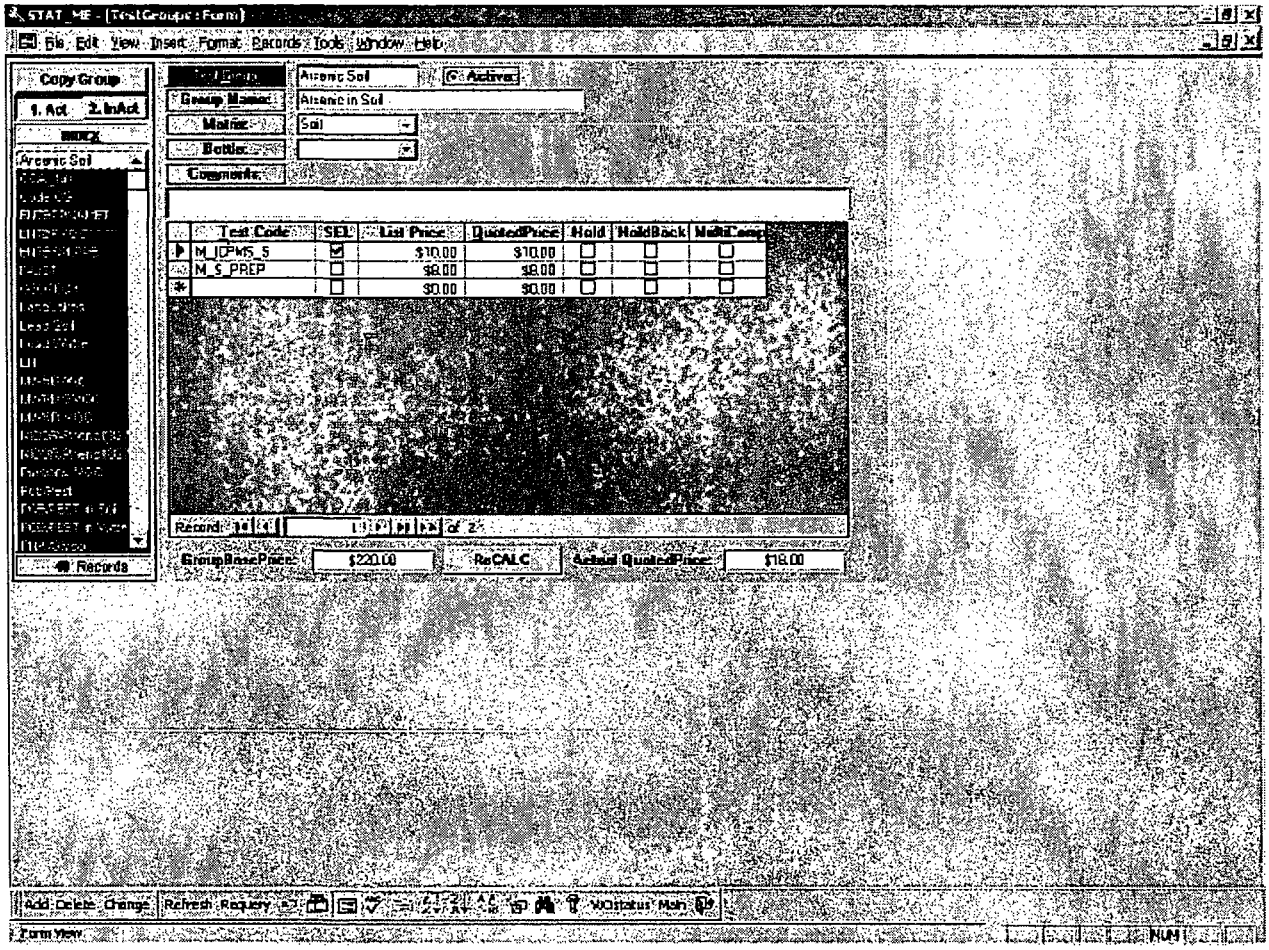

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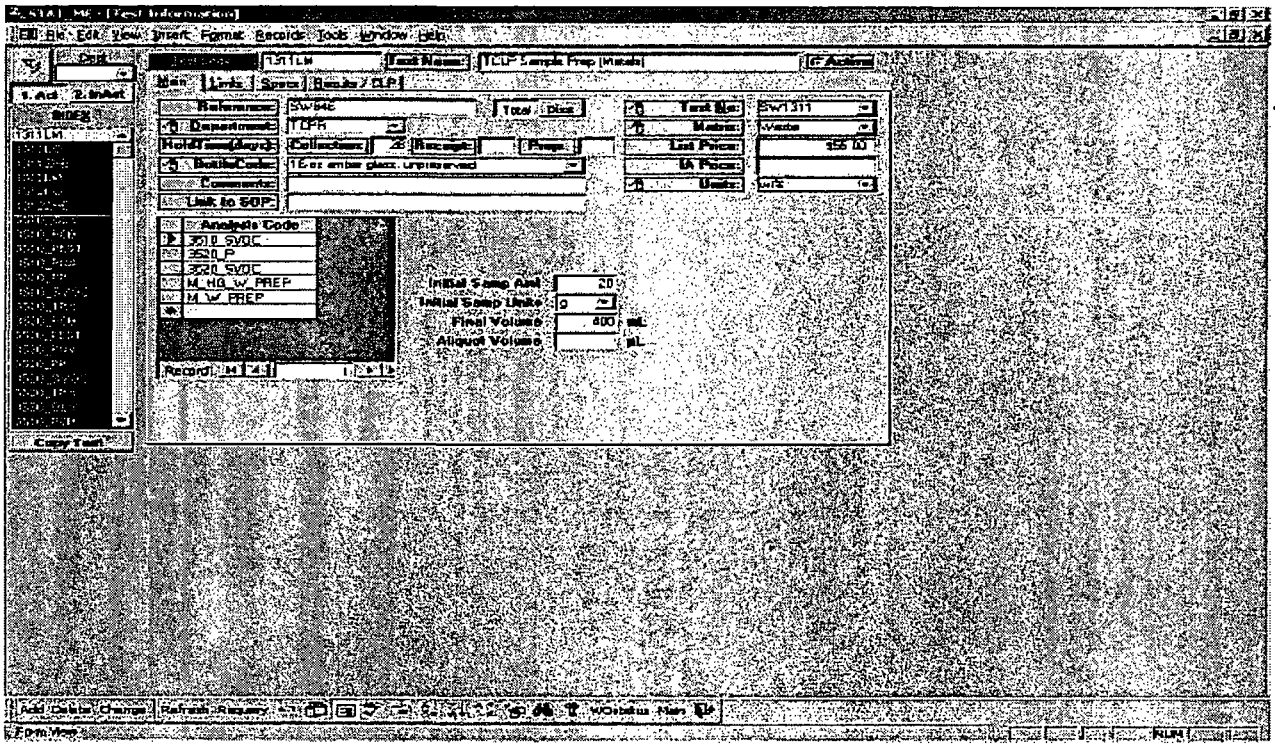

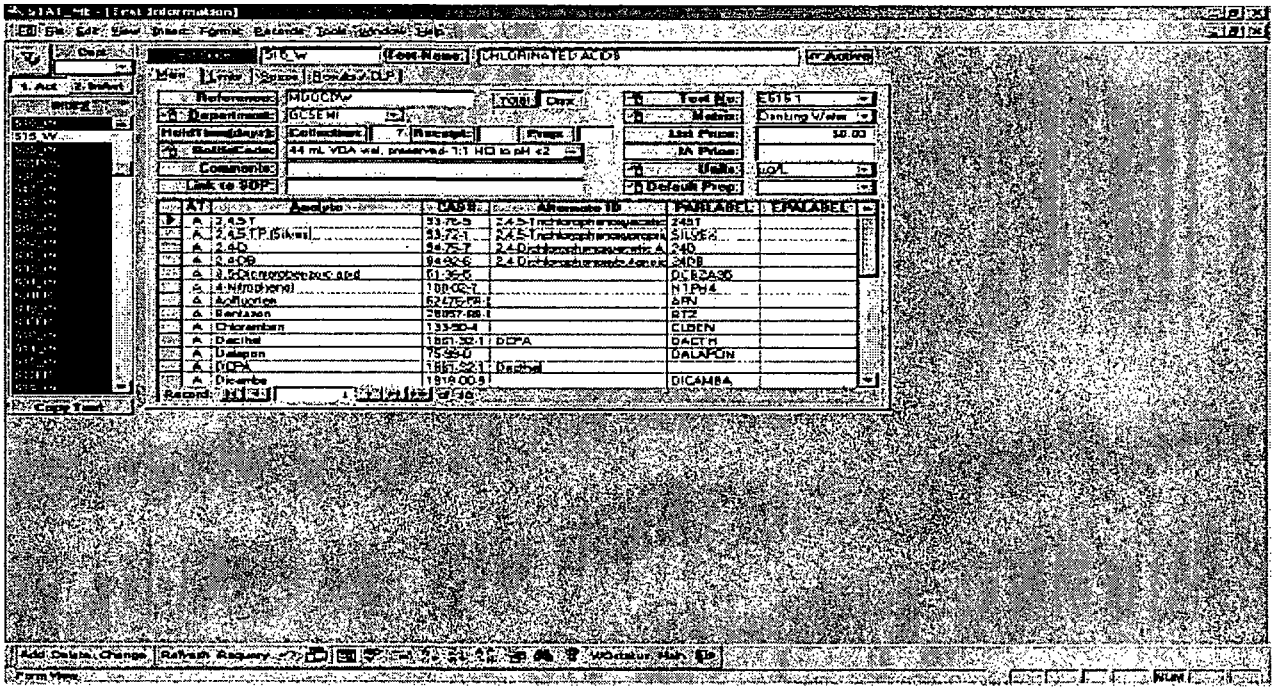

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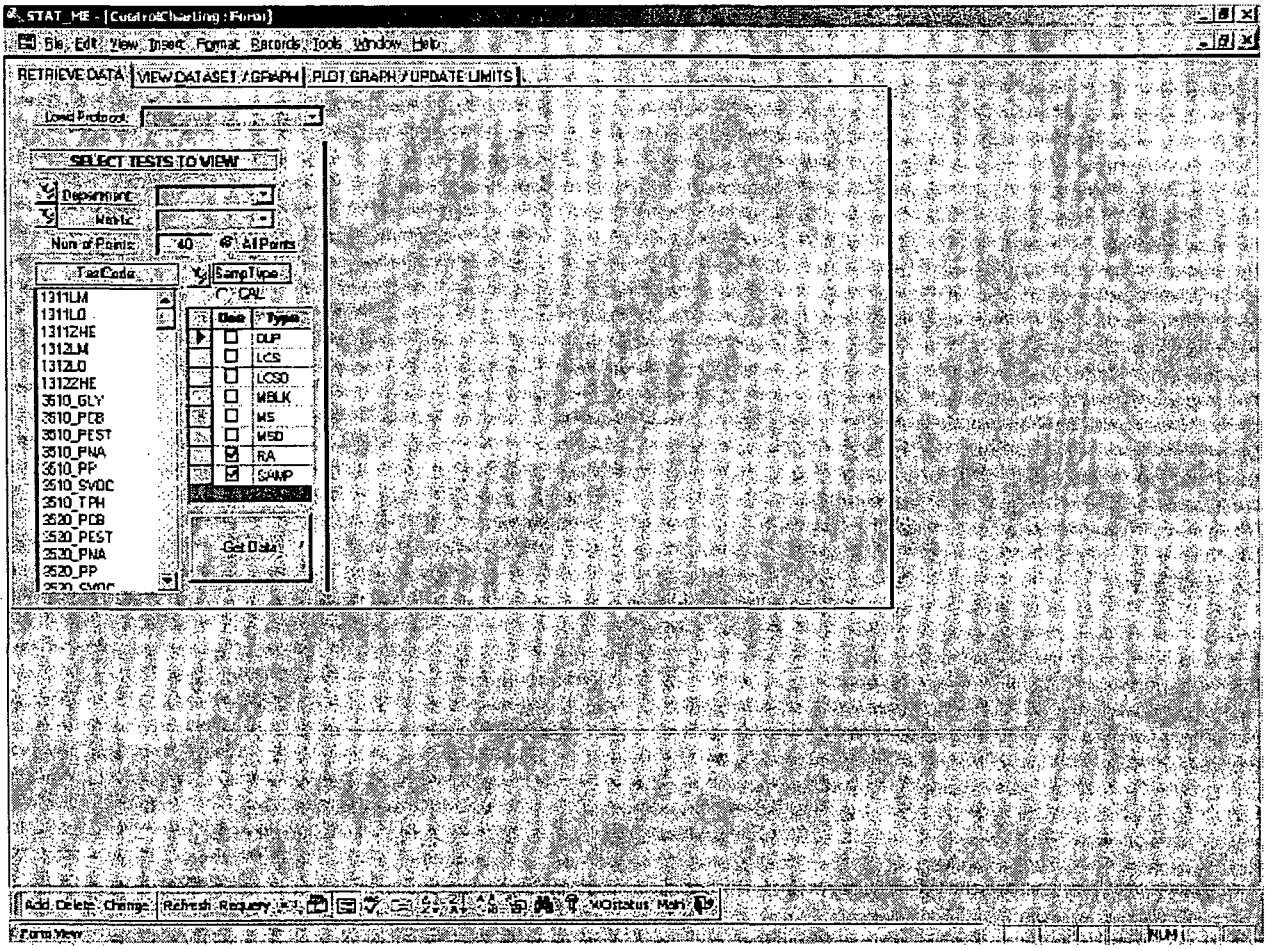

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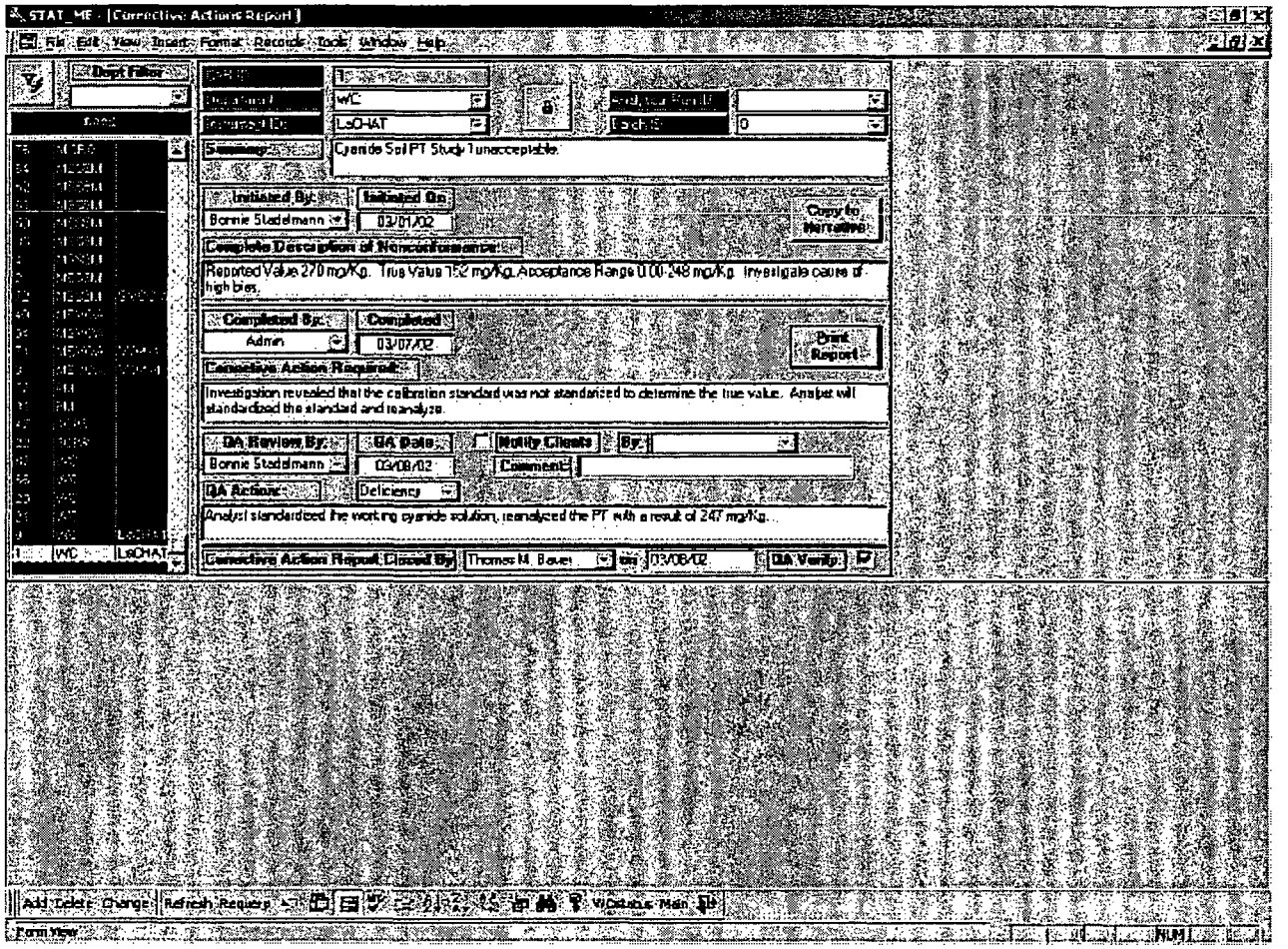

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 3005:

SW846 3005A Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA, ICP or ICP-MS

Revision 01
Effective Date: May 10, 2005

Author: **Dennis Jachim**

Printed Name	Signature/Date
Bruce Anderson **Inorganics Manager**	_____
Dennis Jachim **Technical Manager**	_____
Laurie Fetterman **QA Manager**	_____
Donald Cortes **Laboratory Director**	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

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Table of Contents

1. Identification of Test Method

SW846 3005A Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA, ICP, or ICP-MS.

2. Applicable Matrix or Matrices

This method is applicable to the following matrices: waters, and SPLP and TCLP extracts.

3. Detection Limits

Detection limits for this method are not applicable. Please refer to the analytical SOP 4510 Metals Analysis by Inductively Coupled Plasma-Mass Spectrometry and SOP 4550 Analysis of Lead by atomic Absorption Direct Aspiration for the corresponding detection limits.

4. Scope and Application

4.1 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by FLAA, ICP or ICP-MS. The procedure is used to determine total recoverable metals.

Samples prepared by this SOP may be analyzed for the following:

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Boron	Selenium
Cadmium	Silver
Calcium	Sodium
Chromium	Thallium
Cobalt	Tin
Copper	Titanium
Iron	Vanadium
Lead	Zinc

4.2 When analyzing for total dissolved metals, filter the sample at the time of collection, prior to acidification with nitric acid.

4.3 This method is restricted to use by or under the supervision of analysts experienced in the digestion process. Each workcell/ analyst must demonstrate the ability to generate acceptable results with this method.

5. Summary of Test Method

5.1 Total recoverable metals – The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substantially reduced in volume. The digestate is filtered (optional) and diluted to volume and is then ready for analysis.

5.2 Dissolved metals – The sample is filtered through a 0.45 µm filter. Samples for dissolved metals do not need to be digested as long as the acid concentrations have been adjusted to the same concentration as in the standards.

5.3 *Method Modifications*

5.3.1 The laboratory uses this procedure to digest samples for ICP-MS analysis.

5.3.2 The laboratory uses slightly different concentrations of acids to perform the digestion due to the use of ICP-MS.

5.3.3 The laboratory uses this preparation procedure for the analysis of boron, tin and titanium. These analytes are not originally covered in the scope of EPA 3005A.

6. Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7. Interferences

The analyst should be cautioned that this digestion procedure might not be sufficiently vigorous to destroy some metal complexes.

8. Safety

8.1 General laboratory protection (safety glasses, lab coat, disposal latex/nitrile gloves) should be worn at all times when handling samples or reagents. Use extreme caution when handling concentrated acids.

8.2 The digestion, and all reagent additions must be performed in a fume hood.

8.3 The potential health hazards from the samples cannot be determined. Therefore, exposure to the samples should be minimized to the extent possible.

8.4 Other safety precautions must be conducted in accordance with the Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of material safety data sheets (MSDS) is available in each room for personnel involved in an analysis using chemicals.

9. Equipment and Supplies

9.1 The following apparatus is recommended for performing this procedure. Equivalent items can be used, if with their use, the analytical and QA/QC requirements in this SOP can be met.

9.1.1 Polypropylene Digestion Vessels or equivalent, graduated 70 mL

9.1.2 25 mm Polypropylene ribbed watch glasses, or equivalent.

9.1.3 Thermometer capable of measuring to at least 125°C with suitable precision and accuracy.

9.1.4 Filter paper – Whatman No. 41 or equivalent.

9.1.5 0.45 micron filter disks (Whatman Paradisc (13mm)) and 10 mL syringe (used for dissolved metals analysis)

9.1.6 Disposable tubes, approximately 15 mL

9.1.7 CPI ModBlock block digestor or equivalent – Adjustable and able to maintain a temperature of 90-100°C.

9.1.8 Funnel or equivalent.

9.1.9 Autopipetter: 0.010 to 0.10 mL, 0.10 to 1.0 mL, 1.0 to 5.0 mL

9.1.10 Acid dispensers, repipet 1-10 mL, 1-5 mL

9.1.11 pH paper, wide range

9.1.12 Graduated cylinder – 1 Liter

9.1.13 150 mL glass Pyrex® Griffin beakers or equivalent

9.1.14 40 to 50 mm glass ribbed watch glass

9.1.15 Thermolyne Cimarec Model 3 hot plate or equivalent

10. Reagents and Standards

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see SOP 230 Corrective Action). Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is questionable, analyze the reagent to determine the level of impurities. The reagent blank must be less than the RL in order to be used.

10.1 Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagent Receipt and Preparation.

10.2 Reagents – In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used.

10.3 Reagent Water - Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified. An in-house water system is used.

10.4 Nitric acid (concentrated), HNO_3 – Trace Metal Grade, and analyzed to determine level of impurities. If method blank is < RL, the acid can be used.

10.5 Nitric Acid 1:1 – Place 20 Liter carboy in sink. Add approximately 9 Liters of reagent water. Slowly add Nitric Acid (4-2.5 Liter bottles) to the carboy at a rate of one bottle every minute. Bring up to the 20 Liter mark with reagent water. Mix well. ***Note: Lab coats, gloves, safety glasses, and a face shield must be worn when preparing the 1:1 Nitric Acid.***

10.6 Hydrochloric acid (concentrated), HCL - Trace Metal Grade, and analyzed to determine level of impurities. If method blank is < RL the acid can be used.

10.7 ICPMS LCS and Matrix Spiking Solution: Spike 1 mL of the working spike solution per 50 mL of sample.

10.8 FLAA Spike Solution - Spike 0.5 mL of the lead ICV/CCV stock intermediate per 50 mL of sample.

11. Sample Collection, Preservation, Shipment and Storage

All samples are stored away from all standards, reagents, food and other potentially contamination sources in order to prevent cross contamination.

11.1 Sample Collection: 250 mL or 500 mL plastic bottle

11.1.1 Total recoverable metals – All samples must be acidified at the time of collection with HNO_3 (~ 5 mL/L) to pH <2.

11.1.2 Dissolved metals – All samples must be filtered through a 0.45-µm filter and then acidified at the time of collection with HNO_3 (~ 5 mL/L) to pH <2. (If the unpreserved sample is filtered in the laboratory, a blank and a laboratory control sample must also be filtered, and analyzed with the batch.)

11.2 Sample Storage: Samples may be chilled to 0.1 - 6°C or stored at room temperature.

11.3 Digestate Storage: store at room temperature

11.4 Holding time is 180 days from time of sample collection.

12. 0 Quality Control

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality.

The results of these QCI samples are used to assess the acceptability of data.

12.1 Blanks

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method blank shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second blank shall be prepared after the twentieth sample. The method blank shall be processed through all preparatory steps used for the samples, including cleanup procedures. The blank shall be analyzed using the same instrument and conditions as the samples.

12.2 Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second LCS shall be prepared after the twentieth sample. The LCS shall be processed through all preparatory steps used for the samples, including cleanup procedures. The LCS shall be analyzed using the same instrument and conditions as the samples. Refer to Section 10.7 for instructions.

12.3 Duplicates

Duplicates of field samples or of the LCS must be prepared in compliance with the method requirements and client directives. Note: the analysis of the Matrix Spike Duplicate (MSD) is used as a substitute for the laboratory duplicate. In those cases when there is insufficient sample to perform either a duplicate analysis or MSD analysis, the duplicate analysis of the LCS (LCS/LCSD) is used to judge the precision of the analytical results.

12.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire batch. MS/MSDs must be analyzed at a minimum of 1 per 20 samples per matrix per preparation procedure, or as specified by the required test method.

Samples chosen for matrix spiking are rotated among different clients and/or different client projects. This is accomplished through communication between the Department Manager and the analyst. In addition, designated samples, as indicated by client request or contract requirement, are matrix spiked.

The MS/MSD pair shall be processed through all preparatory steps used for the samples. They shall be analyzed using the same instrument and conditions as the samples. Refer to Section 10.7 and section 10.8 for sample spiking instructions for ICP-MS and FLAA, respectively. Some clients may require different spiking levels; these specific needs are documented on the request for analysis forms.

13. Calibration and Standardization

Support Equipment

Autopipettes - Check autopipette to ensure standardization is within control limits (see SOP General Laboratory Practices 1040 for Pipette Calibration).

Thermometers – Check the thermometer to ensure the calibration has not expired (see SOP 1040 General Laboratory Practices for Thermometer Calibration). Check the thermometer during the analysis and record the corrected temperature in the preparation logbook.

14. Procedure

14.1 Block Digestion

14.1.1 Initial Set-up:

14.1.1.1 Turn on power switch

14.1.1.2 Use SELECT button to choose A

14.1.1.3 Push and hold down the ENTER button until OFF appears

14.1.1.4 Select temperature using UP (▲) and DOWN (▼) arrows

14.1.1.5 Push the ENTER button until the status light starts to flash

14.1.1.6 If you want to turn B on, use the SELECT button to choose B and repeat steps 14.1.3 to 14.1.5

14.1.1.7 Status light will stop flashing when the correct temperature is reached

14.1.1.8 Place the dedicated temperature digestion vessel (a vessel filled with water to monitor the temperature of samples) in the block. Allow the temperature to equilibrate. Adjust the thermostat if the temperature is not correct (95°C ± 5°C or 194 to 212°F). Record the corrected temperature reading in the prep logbook.

14.1.2 Transfer an aliquot of well-mixed sample to a sample digestion vessel and fill to the 50-mL mark. Record the sample volume in the prep logbook.

NOTE: All steps requiring the use of acids must be conducted under a fume hood using appropriate laboratory safety equipment.

14.1.3 Add 3 mL of 1:1 HNO_3 and 1 mL of concentrated HCL. Record the reagent identification in the prep logbook. Place the digestion vessel into the block digestor, attach watch glass and heat the sample to 95°C ± 5°C (or 194 to 212°F) until the volume has been reduced to less than one-half (approximately 3 to 6 hours). Record the "start" time in the prep logbook.

CAUTION: Do not boil. Antimony is easily lost by volatilization from hydrochloric acid media.

14.1.4 Remove the vessel and allow it to cool. Wash down the vessel walls and watch glass with water and, when necessary, filter the sample to remove silicates and other insoluble material that could clog the nebulizer. Record the "End Time" in the sample Prep logbook.

14.1.4.1 Filtration using Whatman no. 41 filter paper should be done only if there is concern that insoluble materials may clog the nebulizer; this additional step is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and pre-rinsed with 1:1 HNO_3, followed by reagent water. Filter the entire sample, and rinse the filter with reagent water.

14.1.5 Adjust the final volume to the 50 mL mark with reagent water, cap, and mix well.

14.2 Beaker Digestion

14.2.1 Turn on the hot plate. Place the temperature beaker (a beaker filled with water to monitor the temperature of samples) on the hot plate. Allow the temperature to equilibrate. Adjust the thermostat if the temperature is not correct (95°C ± 5°C or 194 to 212°F). Record the temperature reading in the prep logbook.

14.2.2 Transfer an aliquot of well-mixed sample to a labeled 70-mL polypropylene sample digestion vessel and fill to the 50-mL mark. Record the sample volume in the prep logbook.

14.2.3 Pour the 50 ml of sample into a clean pre-labeled 150 ml beaker. Rinse the vessel and cap and save the labeled 70-mL polypropylene sample digestion vessel.

NOTE: All steps requiring the use of acids must be conducted under a fume hood using appropriate laboratory safety equipment.

14.2.4 Add 3 mL of 1:1 HNO_3 and 1 mL of concentrated HCL. Record the reagent identification in the prep logbook. Place the beaker on the hot plate, cover with a 50-mm glass ribbed watch glass and heat the sample to 95°C ± 5°C (or 194 to 212°F) until the volume has been reduced to less than one-half (approximately 1.5 to 2 hours). Record the "start" time in the prep logbook.

CAUTION: Do not boil. Antimony is easily lost by volatilization from hydrochloric acid media.

14.2.5 Remove the beaker and allow it to cool. Wash down the beaker walls and watch glass with water and, when necessary, filter the sample to remove silicates and other insoluble material that could clog the nebulizer. Record the "End Time" in the sample Prep logbook.

14.2.5.1 Filtration using Whatman no. 41 filter paper should be done only if there is concern that insoluble materials may clog the nebulizer; this additional step is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and pre-rinsed with 1:1 HNO_3, followed by reagent water. Filter the entire sample, and rinse the filter with reagent water.

14.2.6 Transfer the sample back into the labeled 70-mL polypropylene sample digestion vessel. Rinse the beaker with reagent water and bring to a final volume of 50 mL.

15. Data Reduction, Calculations and Loading

15.1 The procedure for uploading data into the LIMS system is detailed in SOP 1400 LIMS.

16. Method Performance

Demonstration of Capability (DOC)

All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

16.1 ICP-MS Metals

A quality control (QC) reference concentrate is required containing elements at an approximate concentration of 10 to 250 µg/L for aqueous samples using the LCS solution (SOP 4510 Metal Analysis by Inductively Coupled Plasma- Mass Spectrometry). Note: The LCS solution may need several different dilutions to accommodate all the analytes. The QC reference sample is made using stock standards prepared independently from those used for calibration.

For each analyte calculate the mean recovery (X) and standard deviation (s) and the average % Recovery (%R). Compare X and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. Note: For aqueous samples, X must be within 10 ± 2 µg/L to 250 ± 50 µg/L and s must be less than 2 and 50 µg/L and %R must be within 100 ± 20%. These limits are taken from established in-house criteria If X and s and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If X or %R falls outside the range for accuracy, or s exceeds the precision limit, then the system performance is unacceptable for that analyte and corrective action must be taken.

16.2 Lead by FLAA

Four Quality Control (QC) reference concentrates are required containing lead at 1 mg/L. They are made (using the LCS solutions and materials in Section 10.9.6) as described in section 12.2 of SOP 4550 Analysis of Lead by Atomic Absorption Direct Aspiration (NIOSH 7082, EPA IO-3.2, and EPA 7420). The QC reference samples are made using stock standards prepared independently from those used for calibration.

Calculate the mean recovery (X) and standard deviation (s) and the average % Recovery (%R). Compare X and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. For aqueous samples, X must be within 1 ± 0.2 mg/L and s must be less than 0.2 mg/L and %R must be within 100 ± 20%. These limits are taken from

established in-house criteria If X and s and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If X or %R falls outside the range for accuracy, or "s" exceeds the precision limit, then the system performance is unacceptable for that analyte and corrective action must be taken.

Comparison to Reference Method Data

Not applicable to this SOP

In-House Control Limits

Not applicable to this SOP

17. Pollution Prevention

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the lab waste disposal program, SOP 1130 Waste Disposal. With the consent of the client, the samples may be returned to their origin for treatment.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory staff is required to protect the laboratory's and our clients' business information when disposing of recycled paper or waste from the facility.

18. Data Assessment and Criteria for Quality Control Measures

The laboratory must maintain records to document the quality of data that is generated. Ongoing quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. The data review is conducted according to SOP 1250 Data Review.

Record any samples not properly preserved, any holding time exceeded, or deviations from this SOP in the logbook.

19. Corrective Actions for Out-Of-Control Data

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action. Corrective action, if necessary, will be implemented by the department manager. Possible actions include:

1) Review standards digestion logbooks. Check all sample data entries and ensure all support equipment checks are properly recorded.
2) Re-Analyze the samples as directed by the department manager.
3) Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the maintenance logbook.

20. Contingencies for Handling Out-Of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director will review data and discusses options with the client. Re-analysis or reporting the data with qualifications are alternatives. Out-of-control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.

20.1 The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

20.2 Client Requested Modifications: See applicable analytical SOP.

21. Waste Management

The STAT Analysis Corporation SOP 1130 Waste Disposal identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded accordance with SOP 1130 Waste Disposal.

22. References

22.1 Method 3005A, U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" Update III, December 1996.
22.2 STAT Analysis Corporation Quality Assurance Manual
22.3 STAT SOP 003 Chemical Hygiene Plan
22.4 STAT SOP 230 Corrective Action
22.5 STAT SOP 1000 Control and Use of Laboratory Notebooks
22.6 STAT SOP 1010 Analytical Standards and Reagent Receipt and Preparation
22.7 STAT SOP 1020 Laboratory Glassware Cleaning
22.8 STAT SOP 1040 General Laboratory Practices
22.9 STAT SOP 1130 Waste Disposal
22.10 STAT SOP 1210 Method Detection Limits
22.11 STAT SOP 1230 Training
22.12 STAT SOP 1250 Data Review
22.13 STAT SOP 1400 LIMS
22.14 STAT SOP 4510 Metals Analysis by Inductively Coupled Plasma- Mass Spectrometry
22.15 STAT SOP 4550 Analysis of Lead by Atomic Absorption Direct Aspiration (NIOSH 7082, EPA IO-3.2, and EPA 7420)

23. Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

Attachment 1: ICP-MS Preparation Logbook
Attachment 2: Pb Preparation Logbook

ATTACHEMNT 1: ICP-MS Preparation Logbook

Logbook # 23-0007

STAT ANALYSIS

ICP-MS Prep Logbook

Start Time:

End Time:

Temperature:

Thermometer ID#:

Matrix: W = Water, S = Soil, T = TCLP, D = Dissolved

Sp = SPLP, Wp = Wipe, A = Air

Analyst	Date	Sample #	Bottle #	Matrix	Sample Wt./Vol. (g or mls)	Final Volume (mls)	Spike Volume (mls)	Spike Solution ID#	Comments

Reagent ID#s

HNO_3: _____

H_2O_2: _____

HCl: _____

Reviewed by /Date: _____

Attachment 2: Pb Preparation Logbook

Logbook22-0019

STAT ANALYSIS

Lead Prep Logbook

Matrix A = Air, Wp = Wipes, S = Soil, T= TCLP, P= Paint, WW= Waste Water, AA = Ambient Air

Analyst(s)	Date	Start time	End time	Client	Sample #	Count	Matrix	Sample Wt/ Vol (g or mls)	Final Vol (mls)	Spike Amount (g or mls)	Spike Solution ID#	Temp °C	Area / Volume	TAT	Comments

Acid Reagent #: _____ Page 1 of 200 Reviewed by/Date: _____

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 3250

AMMONIA DISTILLATION

Revision 00
Effective Date: November 26, 2008

Printed Name	Signature/Date
Bruce Anderson **Inorganics Manager**	_____
Dennis Jachim **Technical Manager**	_____
Pinaki Banerjee **QA Director**	_____
Donald Cortes **Laboratory Director**	_____

This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

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Table of Contents

1.0 Identification of Test Methods

SOP Title: Ammonia Distillation by 4500-NH_3 B, is also known as Ammonia Distillation in the laboratory records.

2.0 Applicable Matrix or Matrices

This method is used to prepare samples for the determination of the concentration of Ammonia as N in aqueous samples, soils, wastes, and leachates. This method employs a distillation procedure that is used to isolate ammonia compounds from the sample matrix. This distillation method is not applicable to oil or multiphasic samples or samples not amenable to the distillation procedure.

3.0 Detection Limits

The lab follows the procedure found in 40CFR Part 136B to determine the MDL for each matrix type on an annual basis. See the STAT Analysis SOP 1210 for the MDL procedure, frequency and acceptance criteria. The MDLs measured by the lab and all supporting documentation are in the laboratory QA files for review.

The laboratory determined MDL must always be less than the reporting limit (RL). The RL will usually range from three to ten times the laboratory measured MDL but this relationship may vary dependent on dilution of sample aliquots, matrix interferences, moisture adjustments (in solid samples), or method-specified requirements.

See the STAT Analysis SOP 4250 for RL and MDL for ammonia analysis.

4.0 Scope and Application

This method, based on Standard Methods 4500-NH_3 B, is a distillation procedure used to extract ammonia and substituted ammonia from aqueous solutions, solid waste materials, or effluents. For the determination of the concentration of ammonia as N in the distillate refer to SOP 4250 Ammonia Analysis.

5.0 Summary of Method

The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is distilled into an acid solution. The distillate is collected and the ammonia concentration is then determined colorimetrically or by titration. The quantitative analysis of ammonia in the distillate is performed using the analytical procedure detailed in STAT SOP 4250 Ammonia Analysis.

Method Modifications from Reference: STAT Analysis Corporation utilizes a Midi-Still™ Distillation Apparatus. This involves using a smaller sample volume thus producing less waste. A volume of 50 mL is distilled (section 14.2 of this SOP).

6.0 Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7.0 Interferences

7.1 Interferences are eliminated or reduced by using the distillation procedure.

7.2 The samples should be buffered to a pH of 9.5 with a borate buffer in order to decrease hydrolyses of cyanates and organic nitrogen compounds which may decompose under test conditions to generate ammonium ion.

7.3 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of sodium arsenite. If chlorine is not removed, the ammonia compounds may be partially oxidized and the results may be low.

7.4 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

8.0 Safety

8.1 All samples must be assumed as hazardous and appropriate precautions taken during handling.

8.2 Safety glasses, gloves, lab coats and closed toe shoes are to be worn.

8.3 Care must be taken to ensure that the distillation apparatus is a closed system prior to the addition of reagents.

8.4 Other safety precautions must be conducted in accordance with the Chemical Hygiene Plan. Other actions can also be applied, if deemed necessary. A reference file of material safety data sheets (MSDS) is available in the laboratory for personnel involved in any analysis using chemicals.

8.5 The following chemicals have the potential to be highly toxic or hazardous, for a detailed explanation, consult the MSDS.

8.5.1 Sulfuric acid is a strong oxidizer. Use extreme caution when handling. Avoid eye and skin contact. Wash exposed areas immediately with copious amounts of water.

8.5.2 Sodium Hydroxide is a strong reducing agent. Use extreme caution when handling. Avoid eye and skin contact. Wash exposed areas immediately with copious amounts of water.

8.5.3 Sodium Arsenite is toxic. Wear gloves and use in a fume hood. Do not inhale dust.

9.0 Equipment and Supplies

9.1 Reliance Midi-Still™ Midi-Distillation System (or equivalent)
- 9.1.1 MIDI-STILL Ammonia Distillation System – 10 place
- 9.1.2 Distilling Head – Set of 10
- 9.1.3 Condenser, Cold Finger –Set of 10
- 9.1.4 Dispersion/Absorber Tube, Fritted – Set of 10
- 9.1.5 Purge Tube. Reaction Flask – Set of 20
- 9.1.6 Teflon Tubing Set

9.2 Cold water source for condensers

9.3 Top loading balance capable of weighing to 0.01 g

9.4 Centrifuge Tubes, 50 mL graduated

9.5 Volumetric Flasks, Class A: 1000mL, 250mL, 100mL, 50ml, 25mL with stoppers

9.6 Autopipetter: 0.010 to 0.10 mL, 0.10 to 1.0 mL, 1.0 to 5.0 mL

9.7 Glass Beakers, 500mL, 250mL

9.8 pH paper or strips, wide range

9.9 Potassium iodide starch paper

9.10 Test Tubes, 15 mL and Tube Racks

9.11 Plastic and glass bottles for solution storage

10.0 Reagents and Standards

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see SOP 230 Corrective Action).

Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagents Receipt and Preparation.

10.1 **Reagents** – In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. Unless otherwise indicated, all reagents are stored at room temperature for up to 1 year after preparation. Store in a glass or plastic bottles. Use ammonia-free reagent water (1 megaohm) for all solutions.

10.2 **Reagent 1. Distillation Reagent, 1 N Sodium Hydroxide Solution (stock):** In a 500-mL volumetric flask, dissolve 20 g Sodium Hydroxide (NaOH for Nitrogen Determination grade) in 250 mL reagent water, cool and dilute to mark with reagent water.

10.3 **Reagent 2. Distillation Reagent, 3 N Sodium Hydroxide Solution:** In a 100-mL volumetric flask, dilute 12 g Sodium Hydroxide (NaOH for Nitrogen Determination grade) in 70 ml reagent water and dilute to mark with reagent water. Invert to mix.

10.4 **Reagent 3. Sodium Tetraborate solution, 99%:** In 1-liter volumetric flask dissolve 5.0 g anhydrous $Na_2B_4O_7$ or (9.5 g $Na_2B_4O_7$?10 H_2O) in 500 ml of reagent water and dilute to mark with reagent water.

10.5 **Reagent 4. Sodium Arsenite, 0.1 N:** In 100-ml volumetric flask, dissolve 1.3 g $NaAsO_2$ in 100 ml reagent water (used to remove oxidizing agents in samples).

10.6 **Reagent 5. Borate Buffer:** In 1-liter volumetric flask, mix 88 ml 0.1 N Sodium Hydroxide (Reagent 2), 4.75 g $Na_2B_4O_7$ and dilute to 1 liter with reagent water.

10.7 **Reagent 6. Acid receiving solution, 1% Sulfuric Acid:** To a 1-L volumetric flask add approximately 900 ml reagent water, then add 10 ml concentrated Sulfuric Acid. Dilute to the mark with REAGENT water and invert to mix

10.8 **Reagent 7. Mixed Indicator Solution:** Dissolve 200 mg methyl red indicator in 100 mL 95% ethyl or isopropyl alcohol. Dissolve 100 mg methylene blue in 50 mL ethyl or isopropyl alcohol. Combine solutions. Prepare monthly. Prepared reagent can be commercially purchased.

10.9 **Reagent 8. Indicating Boric Acid Solution:** Dissolve 20 g H_3BO_3 in water, add 10 mL mixed indicator solution, and dilute to 1 L. Prepare monthly.

10.10 Ottawa Sand

10.11 Standards

10.11.1 At least one of the standards must be traceable to a NIST traceable source when available. The manufacturer should include a certificate of analysis for each standard. If one is not provided, contact the manufacturer. Retain all certificates in the designated binder (see SOP 1010 Analytical Standards and Reagents Receipt and Preparation).

10.11.2 Standards must be prepared volumetrically using Class-A volumetric glassware, calibrated pipettes, or gas tight syringes.

10.11.3 Stock ICV/CCV Nitrogen Standard (2^{nd} Source) 1000 mg/L Nitrogen: Commercially purchased. Store per manufacturer's recommendations and shelf life. If shelf life not stated, then this solution may be used for twelve months if stored in the original container at 0.1 - 6^0 C and shows no sign of deterioration.

10.11.4 Intermediate Ammonia Standard for the Phenate Method: (Second source, 100 mg N/L): Dilute 5 ml of Stock ICV/CCV Standard to 50 mL with DI water. This solution may be used for six months if stored at 0.1 - 6^{0}C and shows no sign of deterioration.

10.11.5 LCS and MS solutions: The LCS and Matrix Spike solutions also use the 2nd Source Ammonia standard. Prepare the following solutions fresh daily.

10.11.6 LCS for Phenate Method, 2.5 mg/L or 125 mg/Kg: Add 1.25 ml Intermediate Ammonia Standard (100 mg/L) to 50 ml of reagent water. For soils, spike 1 gram of Ottawa sand with 1.25 ml of the Intermediate Ammonia Standard (100 mg/L), and dilute to 50 mL with reagent water.

10.11.7 LCS for Titrimetric Method: Add 2.5 ml Intermediate Ammonia Standard (100 mg/L) to 50 ml of reagent water.

10.11.8 Matrix Spike-Waters at 2.5 mg/L for Phenate Method: Add 50 ml of sample spiked with 1.25 ml of Intermediate Ammonia Standard (100 mg/L).

10.11.9 Matrix Spike-Waters at 5 mg/L for Titrimetric Method: Add 50 ml of sample spiked with 2.5 ml of Intermediate Ammonia Standard (100 mg/L).

10.11.10 Matrix Spike-For the Phenate Method: Soils at 125 mg/Kg: Spike 1.00 gram of customer sample with 1.25 ml Intermediate Ammonia Standard (100 mg/L) and add 50 ml reagent water.

10.11.11 Matrix Spike-For the Titrimetric Method: Soils at 250 mg/Kg: Spike 2.5 gram of customer sample with 1.25 ml Intermediate Ammonia Standard (100 mg/L) and add 50 ml reagent water M

11.0 Sample Collection, Shipment, Preservation and Storage

11.1 **Sample Collection**

500 ml or 1-liter glass or plastic bottles may be used for sampling and storage of samples. The sample is collected with a minimum of headspace in the container.

11.2 **Sample Preservation**

The samples must be preserved to a pH < 4, using approximately 1 mL of 50% H_2SO_4 per 500 mL sample, and may be held for 28 days at 0.1 - 6°C. Note: If residual chlorine is present, add a few drops of Sodium Arsenite. Test sample using KI starch paper to confirm absence of residual chlorine (a blue color indicates a need for additional treatment). Record the addition of the Sodium Arsenite (amount and chemical ID) in the logbook.

11.3 **Sample Shipment, Handling, and Storage**

Samples shall be placed on ice immediately after collection. The holding time is 28 days for a refrigerated sample (at 0.1 - 6°C) with proper chemical preservation (pH < 4). Distillation and analysis must occur within the 28 days period to be compliant.

12.0 Quality Control

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality.

The results of these QCI samples are used to assess the acceptability of data.

12.1 Blanks

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method blank shall be prepared once per preparation batch of 20 or less samples per matrix type (see Section 6 for definition of a prep batch). If more than 20 samples are prepared a second blank shall be prepared after the twentieth sample. The method blank shall be processed through all preparatory steps used for the samples, including cleanup procedures. The blank shall be analyzed using the same instrument and conditions as the samples.

12.2 Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second LCS shall be prepared after the twentieth sample. The LCS shall be processed through all preparatory steps used for the samples, including cleanup procedures. The LCS shall be analyzed using the same instrument and conditions as the samples. Refer to section 10.11.6 and 10.11.7 for preparation and concentration instructions.

12.3 Duplicates

Duplicates of field samples or of the LCS must be prepared in compliance with the method requirements and customer directives. Note: the analysis of the Matrix Spike Duplicate (MSD) is used as a substitute for the laboratory duplicate. .

12.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire batch. MS/MSDs must be analyzed at a minimum of 1 per 20 samples per matrix per preparation procedure, or as specified by the required test method. For cases where the sample cannot be divided (e.g., wipes, air samples, not enough sample provided by customer) and thus a MS/MSD pair cannot be prepared in the preparation batch, an LCS/LCSD pair is prepared and analyzed to measure precision.

Samples chosen for matrix spiking are rotated among different customers and/or different customer projects. This is accomplished through communication between the Department Manager and the analyst. In addition, designated samples, as indicated by customer request or contract requirement, are matrix spiked.

The MS/MSD pair shall be processed through all preparatory steps used for the samples. They shall be analyzed using the same instrument and conditions as the samples. Refer to

sections 10.11.8, 10.11.9, 10.11.10, and 10.11.11 for preparation and concentration instructions. Some customers may require different spiking levels; these specific needs are documented on the request for analysis forms.

13.0 Calibration and Standardization

Autopipettes - Check autopipette to ensure standardization is within control limits (see SOP 1040 General Laboratory Practices for Pipette Calibration).

Balances - Be sure the balance is checked prior to use and performance criteria are met (see SOP 1040 General Laboratory Practices for Calibration of Balances).

14.0 Procedure

14.1 Sample Pretreatment – Aqueous Samples

14.1.1 Check samples for presence of oxidization agents such as chlorine using KI starch paper. A distinct blue color on the test paper indicates the presents of chlorine. If chlorine is present remove by adding a few drops of Sodium Arsenite Solution (Reagent 4).

14.1.2 Check the pH of aqueous samples using pH paper in order to know how much NaOH to add in 14.2.2.

14.2 Distillation Procedure

14.2.1 Prepare all blanks, LCSs, MS/MSDs and samples in the same manner. All preparations must contain a volume of 50 mL water.

14.2.2 Aqueous samples: Using a 50-mL graduated tube, measure 50 mL of aqueous sample. For samples that require dilution, measure the appropriate volume of sample using a pipette. Add additional reagent water to achieve a 50 mL final volume in the distillation tube. Record the sample ID and volume used for distillation. Adjust pH of the samples to 7, using Reagent 1 or using Reagent 2 when sample is preserved. Caution: **Do not overadjust**. If the pH goes above 7, any ammonia complexes present may be released. If this happens you must restart the pH adjustment with a new aliquot. Add pH adjusted sample to the distillation tubes (Back).

14.2.3 Sediment/soil samples: Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks. Record sample treatment in the logbook if the sample was not homogeneous. Weigh approximately 1.00 g of sample to the nearest 0.01g, and add to the distillation tube (Back). Add 50 mL reagent water into the distillation tube. Record the Sample ID and the weight.

14.2.4 Add to each tube 2.5 ml Borate Buffer (Reagent 5). PH may need to be adjusted to 9.5, after adding borate buffer.

14.2.5 Spike the LCS/LCSD, MS, and MSD, refer to section 10.11.5 – 10.11.8.

14.2.6 Add a few boiling chips to each distillation tube.

14.2.7 To each collection tube add 10 ml Acid Receiving Solution (Reagent 6). For Titrimetric Analysis, add 5 mL. of Indicating Boric Acid Solution (Reagent 8) and 5mL of DI water.

14.2.8 Assemble the remaining glassware apparatus.

14.2.9 Place the distillation tube into the distillation sample tube rack (Back).

14.2.10 Place the collector tube into the collector tube rack (Front).

14.2.11 Place the cold finger condenser into the collector tube with the hose barbs facing toward the rear.

14.2.12 If not already connected, connect the cold finger water tubes to the water manifold with Quick Disconnect adapters to the rear of the unit. The lower tube connects to the forward manifold (water outlet) and the upper tube connects to the rear manifold (water inlet).

14.2.13 Turn on cooling water to condensers, approximately 6 GPH per sample. [For 10 samples set at approximately 60 GPH]

NOTE: **DO NOT** operate the cooling water unless the cold finger condensers are connected to the water manifold. Improper use may damage the cold finger condensers and/or rupture tubing.

14.2.14 Turn on the red rocker switch for main power, the light will glow.

14.2.15 Verify that the operating temperature is set at 150°C.

14.2.16 Collect about 40-50 mL of distillate.

14.2.17 Depress the timer start switch. (Black).

14.2.18 The timer will automatically turn off the heat block after the selected time has expired. Cooling water flow is unaffected.

14.2.19 Allow the samples to cool approximately 15 minutes before turning off cooling water.

14.2.20 Disconnect the tubing couplers.

14.2.21 Remove the collector tube and pour the sample into a pre-labeled 50 mL centrifuge tube. Complete the transfer using an additional 1 to 2 ml of reagent water to rinse the collector tube and to bring the final volume to 50 mL.

14.2.22 Cap the 50 mL tube and shake to mix. If the distillates are not immediately analyzed, refrigerate the distillates at 0.1 – 6 ^{0}C.

14.2.23 Dispose of the used sample from the distillation tubes and wash all the glassware.

14.2.24 Enter Prep Batch with initial volumes (or weights) and final volumes into LIMS (see SOP 1400 LIMS).

14.3 Documentation requirements. Record the following information in the appropriate logbook or data file. Include any deviations from this procedure.

Analyst initials, date [and time if required by the specific project or QAPP] of analysis, sample number or ID, initial sample volume or weight processed, final distillate volume, sample preservation check, QC sample or solution identifier, reagent solutions identifiers, any dilution information, [beginning and ending times of analytical steps if required by the specific project or QAPP], visual observations, and any other information as deemed necessary.

14.4 Routine Maintenance – Record all non-routine maintenance. Daily required maintenance includes the following: check all tubing connections, check all glassware for chips or cracks, and clean the area around the distillation apparatus.

15.0 Data Reduction, Calculations and Loading

Not applicable to the SOP

16.0 Method Performance

Demonstration of Capability (DOC)

Note: Each analyst must demonstrate the ability to generate acceptable results with this method.

All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

A quality control (QC) reference concentrate is required containing ammonia at a concentration of 1-4 times the reporting limit for aqueous and soil samples using the ICV/LCS solution. The QC reference sample is made using stock standards prepared independently from those used for calibration.

For each analyte, calculate the mean recovery (X), standard deviation (s), relative standard deviation (RSD), and the average % Recovery (%R). Compare X and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. Note: RSD must be equal to or less than 20% and the %R must be within 100 ± 20%. These limits are taken from established in-house criteria If RSD and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If RSD or %R falls outside the range for accuracy and precision, then the system performance is unacceptable for that analyte and corrective action must be taken.

Comparison to Reference Method Data
See SOP 4250 Ammonia Analysis for details.

In-House Control Limits
See SOP 4250 Ammonia Analysis for details.

17.0 Pollution Prevention

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the laboratory waste disposal program and applicable waste disposal regulations. With the consent of the customer, the samples may be returned to their origin for treatment.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory Staff are required to protect the laboratory's and our customer's business information when disposing of or recycling waste from the facility.

18.0 Data Assessment and Criteria for Quality Control Measures

The review of the LCS, MS/MSD recovery, and any other QC sample result for acceptable performance for each batch of samples is performed by the analyst performing the analytical procedure and sample analyses. Refer to SOP 4250 Ammonia Analysis, section 18.0 for data assessment and criteria for quality control measures.

19.0 Corrective Action for Out-of-Control Data

The process for handling corrective actions is found in SOP 230 Corrective Action.

If the CCV, MB, LCS/LCSD, MS/MSD, or lab duplicate recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the samples is suspect and is only reported for regulatory compliance purposes with the appropriate corrective action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. Final data results must be qualified in the customer report for reported results not meeting the laboratory-defined criteria.

1) Review standards preparation logbooks. Check all calculations and ensure dilution factors are properly recorded.

2) Re-prepare the suspected standard or QC sample to identify possible preparation errors of the standard or QC sample.

3) Re-Analyze the samples when the CCV or LCS is not within acceptable limits.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

Every effort is made to prevent problems from occurring. When out-of-control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director review the data and discusses options with the customer. Reanalysis or reporting the data with qualification are alternatives. Out-of-control or unacceptable data reported to the customer include the data qualifier, flag and discussion on the rationale for reporting.

The process for handling unacceptable and out-of-control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by the Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21.0 Waste Management

The STAT Analysis Corporation SOP 1130 Waste Disposal identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded accordance with SOP 1130 Waste Disposal.

22.0 References

22.1 Method 350.2, "U.S. EPA National Exposure Research Laboratory (NERL) [formerly EMSL]
22.2 4500-NH_3 B, Standard Methods for the Examination of Water and Wastewater (20th Edition)
22.3 Instruction Manual for MIDI-STIL Midi-Distillation Systems
22.4 STAT Analysis Corporation Quality Assurance Manual
22.5 STAT SAP 003 Chemical Hygiene Plan
22.6 STAT SOP 230 Corrective Action
22.7 STAT SOP 1000 Control and Use of Laboratory Notebooks
22.8 STAT SOP 1010 Analytical Standards and Reagents Receipt and Preparation
22.9 STAT SOP 1020 Glassware Cleaning
22.10 STAT SOP 1040 General Laboratory Practices
22.11 STAT SOP 1130 Waste Disposal
22.12 STAT SOP 1210 Method Detection Limits (MDL's)
22.13 STAT SOP 1230 Training
22.14 STAT SOP 1250 Data Review
22.15 STAT SOP 1400 LIMS
22.16 STAT SOP 4250 Ammonia Analysis

23.0 Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

None.

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 3620

PHENOLICS AAP: DISTILLATION by EPA 9065

Revision 01
Effective Date: June 6, 2005

Authors: Ian Graske and Bruce Anderson

Printed Name	Signature/Date
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Table of Contents

1.0 Identification of Test Methods

SOP Title: Phenolics 4AAP: Distillation by EPA 9065.

2.0 Applicable Matrix or Matrices

This method is used to prepare samples for the determination of the concentration of total phenols in aqueous samples, wastes, leachate, and soils. This method employs a distillation procedure that is used to isolate phenolic compounds from the sample matrix. This distillation method is not applicable to oil or multiphasic samples or samples not amenable to the distillation procedure.

3.0 Detection Limits

Refer to SOP 4715 Automated Phenols –4AAP Analysis by EPA 9066 for MDL's and reporting ranges.

4.0 Scope and Application

This method is a distillation procedure used to extract phenol and substituted phenols from wastes and leachates. For the determination of the concentration of phenols in the distillate refer to SOP 4715 Automated Phenols –4AAP Analysis by EPA 9066.

Note: Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 Summary of Method

Phenols are released from samples by means of a distillation operation under acidic conditions and collected in the distillate. The phenols concentration in the distillate is then determined colorimetrically. The quantitative analysis of phenols in the distillate is performed using the analytical procedure detailed in STAT SOP 4715 Automated Phenols – 4AAP Analysis by EPA Method 9066.

Method Modifications from Reference
STAT Analysis Corporation utilizes a Midi-Phenols Distillation Apparatus. This involves using a smaller sample volume thus producing less waste. A volume of 50 mL is distilled (See Section 14.3 of this SOP).

6.0 Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7.0 Interferences

7.1 Interferences are eliminated or reduced by using the distillation procedure.

7.2 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of < 4 with H_2SO_4 and aerating briefly by stirring.

7.3 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

7.4 Background contamination from plastic tubing and sample containers is minimized by using non-reactive plastic ware (e.g., polypropylene or Duraprene).

7.5 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

8.0 Safety

8.1 All samples must be assumed as hazardous and appropriate precautions taken during handling.

8.2 Safety glasses, gloves, lab coats and closed toe shoes are to be worn.

8.3 Care must be taken to ensure that the distillation apparatus is a closed system prior to the addition of acid.

8.4 Other safety precautions must be conducted in accordance with the Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of material safety data sheets (MSDS) is available in each room for personnel involved in an analysis using chemicals.

8.5 The following chemicals have the potential to be highly toxic or hazardous, for a detailed explanation consult the MSDS.

8.5.1 Phenol is toxic and hygroscopic. Use extreme caution when handling this material.

8.5.2 Strong acids and bases, sulfuric acid and sodium hydroxide, are strong oxidizers. Use extreme caution when handling these materials. Avoid eye and skin contact. Wash exposed areas immediately with copious amounts of water.

9.0 Equipment and Supplies

9.1 Reliance Midi-Still™ Midi-Phenols Distillation System (or equivalent)

9.1.1 MIDI-STILL Phenols Distillation System – 10 place

9.1.2 Distilling Head – Set of 10

9.1.3 Condenser, Cold Finger –Set of 10

9.1.4 Dispersion/Absorber Tube, Fritter – Set of 10

9.1.5 Purge Tube. Reaction Flask – Set of 20
9.1.6 Teflon Tubing Set
9.1.7 Boiling chips

9.2 Water source for condensers

9.3 Top loading balance capable of weighing to 0.01 g

9.4 Centrifuge Tubes, 50 mL graduated

9.5 Volumetric Flasks, Class A: 1000mL, 250mL, 100mL, 50mL, 25mL with stoppers

9.6 Autopipetter: 0.010 to 0.10 mL, 0.10 to 1.0 mL, 1.0 to 5.0 mL

9.7 Glass Beakers, 500mL, 250mL

9.8 pH paper or strips, wide range

9.9 Potassium iodide starch paper

9.10 Test Tubes, 15 mL and Tube Racks

9.11 Plastic and glass bottles for solution storage

9.12 Lead Acetate Paper

10.0 Reagents and Standards

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see SOP 230 Corrective Action).

10.1 Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagents Receipt and Preparation

10.2 Reagents – In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. Unless otherwise indicated, all reagents are stored at room temperature for up to 1 year after preparation. Store in a glass or plastic bottles. Use phenol-free reagent water 1 megohm) for all solutions.

10.3 1 M Sodium Hydroxide Solution (stock): In a 500 mL volumetric flask, dissolve 20 g sodium hydroxide (NaOH) in 250 mL reagent water, cool and dilute to mark with reagent water.

10.4 Distillation Reagent 0.1 M Sodium Hydroxide Solution: In a 100 mL volumetric flask, dilute 10 mL 1 M sodium hydroxide (reagent 1) to mark with reagent water. Invert to mix.

10.5 Distillation Reagent 10% Sulfuric Acid (v/v): In a 100 mL volumetric flask, add 70 mL reagent water, then slowly add 10 mL conc. H_2SO_4. Cool and dilute to mark with reagent water.

10.6 Ferrous ammonium sulfate (used to remove oxidizing agents in samples): In a 500 mL volumetric flask, dissolve 0.55 g ferrous ammonium sulfate in 250 mL reagent water containing 0.5 mL H_2SO_4 and dilute to mark with freshly boiled and cooled reagent water.

10.7 Ottawa sand

10.8 Standards

10.8.1 At least one of the standards must be traceable to a NIST traceable source when available. The manufacturer should include a certificate of analysis for each standard. If one is not provided, contact the manufacturer. Retain all certificates in the designated binder (see SOP 1010 Analytical Standards and Reagents Receipt and Preparation .)

10.8.2 Standards must be prepared volumetrically using Class A volumetric glassware, calibrated micropipettes, or gas tight syringes.

10.8.3 Stock ICV/CCV Phenols Standard (2nd Source) 1000 mg phenol/L: Commercially purchased. Store per manufacturer's recommendations and shelf life. If shelf life not stated, then this solution may be used for twelve months if stored in the original container at 4°C and shows no sign of deterioration.

10.8.4 Intermediate Phenols Spiking Solution: (10 mg phenol /L): Dilute 0.50 mL of Phenols ICV/CCV Stock Standard to 50 mL with reagent water. Prepare this solution fresh monthly and store in a glass stoppered bottle at 4°C.

10.8.5 LCS and MS solutions: The LCS and Matrix Spike solutions use the 2nd Source Phenols standard. Prepare the following solutions fresh daily and store in glass-stoppered bottles.

10.8.5.1. LCS 0.10 mg/L or 5 mg/Kg: Spike with 0.50 mL Intermediate Phenols Standard (10 mg/L) into 50 mL of water or 1 gram of Ottawa sand.

10.8.5.2. Matrix Spike – Waters at 0.10 mg/L: Spike with 0.50 mL Intermediate Phenols Standard (10 mg/L) into 50 mL of sample.

10.8.5.3. Matrix Spike – Soils at 5 mg/Kg: Spike with 0.50 mL Intermediate Phenols Standard (10 mg/L) into 1 gram of Ottawa sand.

11.0 Sample Collection, Shipment, Preservation and Storage

11.1 Sample Collection

500 mL or 1 Liter glass bottles may be used for sampling and storage of samples. The sample is collected with a minimum of headspace in the container.

11.2 Sample Preservation

The water samples must be preserved to a pH < 4, using approximately 1 mL of 50% H_2SO_4 per 500 mL sample, and may be held for 28 days at 4°C. Store soil sample at 0.1 to 6 °C.

Note: If residual chlorine is present, add a few drops of ferrous ammonium sulfate. Test sample using KI starch paper to confirm absence of residual chlorine (a blue color indicates a need for additional treatment). Record the addition of the ferrous ammonium sulfate (amount and chemical ID) in the logbook.

11.3 Sample Shipment, Handling, and Storage
Samples shall be placed on ice immediately after collection. The holding time is 28 days for a refrigerated sample (0.1 to 6 °C.) with proper chemical preservation (pH < 4). Distillation and analysis must occur within the 28-day period to be compliant.

12.0 Quality Control

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality.

The results of these QCI samples are used to assess the acceptability of data.

12.1 Blanks

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method blank shall be prepared once per preparation batch of 20 or less samples per matrix type (see Section 6 for the definition of a preparation batch). If more than 20 samples are prepared a second method blank shall be prepared after the twentieth sample. The method blank shall be processed through all preparatory steps used for the samples, including cleanup procedures. The blank shall be analyzed using the same instrument and conditions as the samples.

12.2 Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second LCS shall be prepared after the twentieth sample. The LCS shall be processed through all preparatory steps used for the samples, including cleanup procedures. The LCS shall be analyzed using the same instrument and conditions as the samples. Refer to section 10.8.5 for preparation and concentration instructions.

12.3 Duplicates

Duplicates of field samples or of the LCS must be prepared in compliance with the method requirements and client directives. Note: the analysis of the Matrix Spike Duplicate (MSD) is used as a substitute for the laboratory duplicate. In those cases when there is insufficient sample to perform either a duplicate analysis or MSD analysis, the duplicate analysis of the LCS (LCS/LCSD) is used to judge the precision of the analytical results.

12.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire batch. MS/MSDs must be analyzed at a minimum of 1 per 20 samples per matrix per preparation procedure, or as specified by the required test method. For cases where the sample cannot be divided (e.g., wipes, air samples, not enough sample provided by customer) and thus a MS/MSD pair cannot be prepared for the preparation batch, an LCS/LCSD pair is analyzed to measure precision and accuracy.

Samples chosen for matrix spiking are rotated among different clients and/or different client projects. This is accomplished through communication between the Department Manager and the analyst. In addition, designated samples, as indicated by client request or contract requirement, are matrix spiked.

The MS/MSD pair shall be processed through all preparatory steps used for the samples. They shall be analyzed using the same instrument and conditions as the samples. Refer to section 10.8.5 for preparation and concentration instructions. Some clients may require different spiking levels; these specific needs are documented on the request for analysis forms.

13.0 Calibration and Standardization

Autopipettes - Check autopipette to ensure standardization is within control limits (see SOP 1040 General Laboratory Practices for Pipette Calibration).

Balances - Be sure the balance is checked prior to use and performance criteria are met (see SOP 1040 General Laboratory Practices for Calibration of Balances).

14.0 Procedure

All sample containers must be labeled with sample ID number. This includes the Reaction Flask, Absorber Tube, and the LACHAT Autosampler 14 ml sample tube. The 50 ml disposable sample tube (and cap) should be labeled with the batch number as well as the sample ID number.

14.1 Sample Pretreatment – Aqueous Samples

14.1.1 Check samples for presence of oxidization agents such as chlorine using KI starch paper. A distinct blue color on the test paper indicates the presents of chlorine. If chlorine is present remove as in 11.2 and note it in the logbook.

14.1.2 Adjust the sample pH to approximately 4 with a few drops of 0.1 M NaOH (or a few drops of 10% H_2SO_4 if the sample was not pre-preserved). Note in the logbook if the sample was not received with a pH < 4.

14.2 Distillation Procedure

14.2.1 Prepare all blanks, LCSs, MS/MSDs and samples in the same manner. All preparations must contain a volume of 50 mL water.

14.2.2 Aqueous samples: Using a 50 mL graduated tube, measure 50 mL of aqueous sample and add to the distillation tubes (Back) with a few boiling chips. For samples that require dilution, measure the appropriate volume of sample using a pipette. Add additional reagent water to achieve a 50 mL final volume in the distillation tube. Record the sample ID and volume used for distillation.

14.2.3 Sediment/soil samples: Mix sample thoroughly, especially composited samples and add a few boiling chips. Discard any foreign objects such as sticks, leaves, and rocks. Record sample treatment in the logbook if the sample was not homogeneous. Weigh approximately 1.0 g sample and add to the distillation tube (Back). Add 50 mL reagent water and 1 drop of 10% H_2SO_4 into the distillation tube. Record the Sample ID and the weight to the nearest 0.01g.

14.2.4 Assemble the remaining glassware apparatus.

14.2.5 Place the distillation tube into the distillation sample tube rack (Back).

14.2.6 Place the collector tube into the collector tube rack (Front)

14.2.7 Place the cold finger condenser into the collector tube with the hose barbs facing to the rear.

14.2.8 IF not already connected, connect the cold finger water tubes to the water manifold Quick Disconnect adapters to the rear of the unit. The lower tube connects to the forward manifold (water outlet) and the upper tube connects to the rear manifold (water inlet).

14.2.9 Turn on cooling water to condensers, approximately 6 GPH/ sample. [For 10 samples set at approximately 60 GPH]

NOTE: **DO NOT** operate the cooling water unless the cold finger condensers are connected to the water manifold. Improper use may damage the cold finger condensers and/or rupture tubing.

14.2.10 Turn on the red rocker switch for main power and the light will glow.

14.2.11 Verify that the operating temperature is set at 180°C.

14.2.12 Verify that the timer is set to 1-¾ hours (105 minutes). This time setting allows for 15 minutes heat up time and 90 minutes of distillation time. This will allow about 40 mL to be collected in the tube. If the tube reaches a volume of 40 mL prior to the end of the timed period, disconnect the tube. Record the start time in the logbook. At the end of the time period, check the block to ensure that the timer has worked properly and that the heat is off. Record the stop time. This ensures that the timer is working properly.

14.2.13 Depress the timer start switch. (Black).

14.2.14 The timer will automatically turn off heat block after selected time has expired. Cooling water flow is unaffected.

14.2.15 Allow the system to cool for approximately 15 minutes before turning off cooling water.

14.2.16 Disconnect the tubing couplers.

14.2.17 Remove the collector tube and pour the sample into a prelabeled 50 mL centrifuge tube. Complete the transfer using additional 1 to 2 mL of reagent water to rinse the collector tube and to bring the final volume to 50 mL.

14.2.18 Cap the 50 mL tube and shake to mix. If the distillates are not immediately analyzed, refrigerate the distillates at 4°C.

14.2.19 Dispose of the used sample from the back tubes and wash all the glassware.
14.2.20 Enter Prep Batch with initial volumes (or weights) and final volumes into LIMS (see SOP 1400 LIMS).

14.3 Documentation requirements. Record the following information in the appropriate logbook or data file. Include any deviations from this procedure.

Analyst initials, date [and time if required by the specific project or QAPP] of analysis, sample number or ID, initial sample volume or weight processed, final distillate volume, sample preservation check, QC sample or solution identifier, reagent solutions identifiers, any dilution information, [beginning and ending times of analytical steps if required by the specific project or QAPP], visual observations, and any other information as deemed necessary.

14.4 Routine Maintenance – Record all maintenance. Daily required maintenance includes the following: check all tubing connections, check all glassware for chips or cracks, and clean the area around the distillation apparatus.

15.0 Data Reduction, Calculations and Loading

Not applicable to the SOP

16.0 Method Performance

Demonstration of Capability (DOC)

All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

A quality control (QC) reference concentrate is required containing phenols at a concentration of 0.02 mg/L for aqueous samples and 1 mg/Kg for soil samples using the ICV/LCS solution. The QC reference sample is made using stock standards prepared independently from those used for calibration.

For each analyte calculate the mean recovery (x) and standard deviation (s) and the average % Recovery (%R). Compare x and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. Note: For aqueous samples, x must be within 0.02 ± 0.004 mg/L and s must be less than 0.004 mg/L and %R must be within 100 ± 20%. Note: For soil samples, x must be within 1.0 ± 0.2 mg/Kg and s must be less than 0.2 mg/Kg and %R must be within 100 ± 20%. These limits are taken from established in-house criteria If x and s and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If x or %R falls outside the range for accuracy, or s exceeds the precision limit, then the system performance is unacceptable for that analyte and corrective action must be taken.

Comparison to Reference Method Data

See SOP 4715 Automated Phenols –4AAP Analysis by EPA 9066

In-House Control Limits
See SOP 4715 Automated Phenols –4AAP Analysis by EPA 9066

17.0 Pollution Prevention

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the laboratory waste disposal program and applicable waste disposal regulations. With the consent of the client, the samples may be returned to their origin for treatment.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory staff are required to protect the laboratory's and our clients' business information when disposing of recycling or waste from the facility.

18.0 Data Assessment and Criteria for Quality Control Measures

The review of the LCS result, MS/MSD recovery, and any other QC sample result for acceptable performance for each batch of samples is performed by the analyst performing the analytical procedure and sample analyses. Refer to SOP 4715 Automated Phenols –4AAP Analysis by EPA 9066 Section 18.0 for data assessment and criteria for quality control measures.

19.0 Corrective Action for Out-of-Control Data

The process for handling corrective actions is found in SOP 230 Corrective Action.

See SOP 4715Automated Phenols –4AAP Analysis by EPA 9066 for guidance on handling out-of-control QC.

Review standards preparation logbooks. Check all calculations and ensure dilution factors are properly recorded.

1) Re-prepare the suspected standard or QC sample to identify possible preparation errors of the standard or QC sample.
2) Re-Analyze the samples when the CCV or LCS is not within acceptable limits.
3) Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the

holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discusses options with the client. Reanalysis or reporting the data with qualification are alternatives. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.

The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21.0 Waste Management

The STAT Analysis Corporation SOP 1130 Waste Disposal identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded accordance with SOP 1130 Waste Disposal.

22.0 References

22.1 Method 9065, U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" Update III, December 1996
22.2 National Environmental Laboratory Accreditation Conference (NELAC), Current version at date of signing, USEPA Office of Research and Development, Washington, DC EPA600/R-99-068
22.3 STAT Analysis Corporation Quality Assurance Manual
22.4 STAT SOP 003 Chemical Hygiene Plan
22.5 STAT SOP 230 Corrective Action
22.6 STAT SOP 1000 Control and Use of Laboratory Notebooks
22.7 STAT SOP 1010 Analytical Standards and Reagents Receipt and Preparation
22.8 STAT SOP 1020 Laboratory Glassware Cleaning
22.9 STAT SOP 1040 General Laboratory Practices
22.10 STAT SOP 1130 Waste Disposal
22.11 STAT SOP 1210 Method Detection Limits
22.12 STAT SOP 1230 Training
22.13 STAT SOP 1250 Data Review
22.14 STAT SOP 1400 LIMS
22.15 STAT SOP 4715 Automated Phenols --4AAP Analysis by EPA 9066
22.16 Instruction Manual for MIDI-STIL Midi-Phenols Distillation Systems

23.0 Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

Form 1: Phenols Distillation Logbook

FORM 1: Phenol Distillation Logbook

STAT Analysis Corporation

Logbook #: 37-0003

Phenols Preparation Logbook (9066)

Analyst	Date	Sample ID	Bottle #	KI	Oxidizers	pH	Matrix	Sample Vol/Wt (mL / g)	Final Vol (mL)	Comments

LCS/LCSD/MS/MSD = 0.5 mL 10.0 mg/L Phenol STD

Reviewed by/ Date: _____

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 4250

AMMONIA ANALYSIS

Revision 00
Effective Date: November 26, 2008

Printed Name	Signature/Date
Bruce Anderson **Inorganics Manager**	_____
Dennis Jachim **Technical Manager**	_____
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Donald Cortes **Laboratory Director**	_____

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Table of Contents

1.0 Identification of Test Method

SOP Title: Ammonia Analysis is also known as Ammonia in the laboratory records.

2.0 Applicable Matrix or Matrices

This method is used to determine the concentration of Ammonium ion in aqueous samples, wastes, and leachates. This method is used to quantify the concentration of ammonia from the distillation procedure detailed in STAT SOP 3250 Ammonia Distillation by 4500-NH_3 B

3.0 Detection Limits

The lab follows the procedure found in 40CFR Part 136B to determine the MDL for each matrix type on an annual basis. See the STAT Analysis SOP 1210 for the MDL procedure, frequency and acceptance criteria. The MDLs measured by the lab and all supporting documentation are in the laboratory QA files for review.

The laboratory determined MDL must always be less than the reporting limit (RL). The RL will usually range from three to ten times the laboratory measured MDL but this relationship may vary dependent on dilution of sample aliquots, matrix interferences, moisture adjustments (in solid samples), or method-specified requirements.

For the Phenate Method, the applicable range for aqueous samples is 0.05 to 5 mg/L and the applicable range for soil samples is 2.5 to 250 mg/Kg (as received basis).

The applicable reporting level for titration of aqueous samples is 1 mg/L and for soils is 50 mg/kg (as received basis).

Sample distillates with concentrations greater than the highest calibration standard are diluted and then reanalyzed. Samples with high concentrations of ammonia may also be redistilled using a smaller sample size and then analyzed.

4.0 Scope and Application

The method, based on Standard Methods 4500-NH_3 H and C, is designed for the analysis of aqueous distillates for ammonia. Samples and QC samples are distilled prior to analysis. The distillation procedure described in SOP 3250 Ammonia Distillation by 4500-NH_3 is designed for the determination of ammonia in aqueous solutions, solid waste materials, or effluents. This method is not applicable to oil or multiphasic samples or samples not amenable to the distillation procedure.

This method may be performed using an Automated Phenate Method or the Titrimetric Method. For the automated method, it is restricted to use by or under the supervision of analysts experienced in the use of the Lachat Auto Analyzer.

5.0 Summary of Method

The Automated Method is based on Berthelot reaction of the distillate with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is

added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration in the samples.

The Titrimetric Method is based on titration with sulfuric acid until indicator turns a pale lavender.

Method Modifications from Reference
This SOP reflects the reduced volume version of the method. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.

Degassing to remove air bubbles is performed only if it is noticed that there are air spikes during analysis by the Phenate Method.

6.0 Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7.0 Interferences

7.1 Interferences are eliminated or reduced by using the distillation procedure described in SOP 3250 Ammonia Distillation by 4500-NH_3 B.

7.2 Calcium and Magnesium ions may precipitate if present in sufficient concentration. EDTA (Ethylenediamine Tetraacetate) is added to the sample in line in order to prevent this problem

7.3 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of sodium arsenite. If chlorine is not removed, the ammonium compounds may be partially oxidized and the results may be low.

7.4 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

8.0 Safety

8.1 All samples must be assumed as hazardous and appropriate precautions taken during handling.

8.2 Safety glasses, gloves, lab coats and closed toe shoes are to be worn.

8.3 Other safety precautions must be conducted in accordance with the SAP 003 Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of material safety data sheets (MSDS) is available in the laboratory for personnel involved in an analysis using chemicals.

8.4 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for materials known to be extremely hazardous.

8.5 The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consults the MSDS.

8.5.1 Phenol is toxic and hygroscopic. Use extreme caution when handling this material.

8.5.2 Sulfuric acid is a strong oxidizer and sodium hydroxide is a strong reducing agent. Use extreme caution when handling these materials. Avoid eye and skin contact. Wash exposed areas immediately with copious amounts of water.

8.5.3 Sodium Nitroferricyanide is toxic. Use extreme caution when handling this material. Avoid contact with acids which will releases cyanide gas.

9.0 Equipment and Supplies

9.1 LaCHAT AutoAnalyzer consisting of the following components:

9.1.1 Autosampler Cetac

9.1.2 Reagent Pump

9.1.3 System Unit Lachat 8000

9.1.4 Computer

9.1.5 Printer

9.2 Centrifuge Tubes, 50 mL graduated

9.3 Volumetric Flasks, Class A: 1000mL, 250mL, 100mL, 50mL, 25mL with stoppers

9.4 Autopipetter: 0.010 to 0.10 mL, 0.10 to 1.0 mL, 1.0 to 5.0 mL

9.5 Test Tubes, 15 mL and Tube Racks

9.6 Plastic and glass bottles for solution storage

9.7 Burette: 50 mL. with 0.1 mL graduations

9.8 Beaker: 250 mL.

10.0 Reagents and Standards

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see 230 Corrective Action SOP).

10.1 Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagents Receipt and Preparation.

10.2 Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used.

10.3 Use ammonia-free reagent water (1 Mohm) for all solutions. Degas reagents with helium if necessary to prevent bubble formation.

10.4 **Reagent 1. Sodium Phenolate:** In a 1-liter volumetric flask, dissolve 88 ml of 88% liquefied phenol, or 83 g crystalline phenol in 600 ml reagent water. While stirring, slowly add 32 g Sodium Hydroxide (NaOH for Nitrogen determination), cool, dilute to the mark with reagent water and invert to mix. **Do not degas this reagent.**

10.5 **Reagent 2. Sodium Hypochlorite:** In a 500-ml volumetric flask, mix 109 mL regular Clorox © Bleach (6% sodium hypochlorite) with 125 mL. of DI water. Invert to mix.

10.6 **Reagent 3. Buffer:** In a 1-liter volumetric flask, dissolve 50.0 g Disodium Ethylenediamine Tetraacetate Dihydrate ($Na_2EDTA*2H_2O$) and 5.0 g Sodium Hydroxide (Nitrogen Determination grade) in approximately 900 ml reagent water. Mix until dissolved and dilute to the mark.

10.7 **Reagent 4. Sodium Nitroprusside:** To a 1-liter volumetric flask, dissolve 3.5 g Sodium Nitroprusside (Sodium Nitroferricyanide). Dilute to the mark with reagent water and invert to mix.

10.8 **Reagent 5. Carrier and Diluents (0.20% H_2SO_4):** To a 1-liter volumetric flask, add approximately 900 ml reagent water and 2 ml concentrated Sulfuric Acid. Dilute to the mark with reagent water and invert to mix.

10.9 Reagent 6. 0.02 N Sulfuric Acid (Titrant)

10.10 Standards for Phenate Method

10.10.1 At least one of the standards must be traceable to a NIST traceable source when available. The manufacturer should include a certificate of analysis for each standard. If one is not provided, contact the manufacturer. Retain all certificates in the designated binder (see SOP 1010 Analytical Standards and Reagents Receipt and Preparation).

10.10.2 Standards must be prepared volumetrically using class-A volumetric glassware, calibrated micropipettes, or gas tight syringes. Do not use disposable pipettes to prepare standards.

10.10.3 Nitrogen Stock Calibration Standard: 1000 mg Nitrogen/L: Commercially Purchased. Store per manufacturer's recommendations and shelf life. If shelf life is not stated, then this solution may be used for twelve months if stored in the original container at 0.1 - 6°C and shows no sign of deterioration.

10.10.4 Intermediate Ammonia Calibration Solution: 100 mg Nitrogen/L: Dilute 5 ml of Ammonia Stock Standard to 50 ml with 0.20% H_2SO_4 (Reagent 5). Invert to mix. This solution may be used for six months if stored at 0.1 - 6°C and shows no sign of deterioration.

10.10.5 Stock ICV/CCV Nitrogen Standard (2^{nd}Source) 1000 mg Nitrogen/L: Commercially purchased. Store per manufacturer's recommendations and shelf

life. If shelf life is not stated, then this solution may be used for twelve months if stored in the original container at 0.1 - 6°C and shows no sign of deterioration.

10.10.6 Intermediate (ICV/CCV) Ammonia Standard 100 mg Nitrogen/L: Dilute 5 ml of Stock ICV/CCV Standard to 50 ml with 0.20% H_2SO_4 (Reagent 5), invert to mix. This solution may be used for six months if stored at 0.1 - 6°C and shows no sign of deterioration.

10.10.7 Working ICV STD, 1 mg Nitrogen/L: Dilute 0.5 ml of Intermediate ICV/CCV STD to 50 ml with 0.20% H_2SO_4 (Reagent 5). Prepare this solution fresh daily.

10.10.8 Working CCV STD, 2.5 mg Nitrogen/L: Dilute 1.25 ml of Intermediate ICV/CCV STD to 50 ml with 0.20% H_2SO_4 (Reagent 5). Prepare this solution fresh daily.

10.10.9 The Calibration Standards are prepared according to Table 1 below. Add listed volumes of the Intermediate Ammonia Calibration Standard to each flask, and dilute to mark with 0.20% H_2SO_4 (Reagent 5). Cap and mix will.

Table 1 Ammonia Calibration Standards

Calibration Standard Concentration, mg Nitrogen/L	Amount of Intermediate Calibration Standard mL	Concentration of Intermediate Calibration Standard, mg/L	Final Volume, mL	mg Nitrogen per 50 mL
5	2.5	100	50	0.25
2.5	1.25	100	50	0.125
1	0.5	100	50	0.05
0.5	0.25	100	50	0.025
0.1	0.05	100	50	0.005
0.05	0.025	100	50	0.0025
0.01	0.005	100	50	0.0005
0	0	0	50	0

11.0 Sample Collection, Shipment, Preservation and Storage

Samples shall be placed on ice immediately after collection. The holding time is 28 days for a refrigerated sample (0.1 - 6°C) with proper chemical preservation (pH < 4). Distillation and analysis must occur within the 28 days period to be compliant.

12.0 Quality Control

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality.

The results of these QCI samples are used to assess the acceptability of data.

12.1 Blanks

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method

blank shall be prepared once per preparation batch of 20 or less samples per matrix type If more than 20 samples are prepared a second blank shall be prepared after the twentieth sample. The method blank shall be processed through all preparatory steps used for the samples, including cleanup procedures. The blank shall be analyzed using the same instrument and conditions as the samples.

12.2 Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second LCS shall be prepared after the twentieth sample. The LCS shall be processed through all preparatory steps used for the samples, including cleanup procedures. The LCS shall be analyzed using the same instrument and conditions as the samples.

12.3 Duplicates

Duplicates of field samples or of the LCS must be prepared in compliance with the method requirements and client directives. Note: The analysis of the Matrix Spike Duplicate (MSD) is used as a substitute for the laboratory duplicate.

12.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire batch. MS/MSD's must be analyzed at a minimum of 1 per 20 samples per matrix per preparation procedure, or as specified by the required test method. For cases where the sample cannot be divided (e.g., wipes, air samples, not enough sample provided by customer) and thus a MS/MSD pair cannot be prepared in the preparation batch, an LCS/LCSD pair is prepared and analyzed to measure precision.

The MS/MSD pair shall be processed through all preparatory steps used for the samples. They shall be analyzed using the same instrument and conditions as the samples.

13.0 Calibration For the Phenate Method

Initial Calibration (ICAL)

In addition to achieving the reference method requirements for the minimum number of calibration standards and the acceptance criteria (statistics) for calibration curve fit, the following ICAL criteria also apply:

13.1 The ICAL must be a minimum of 5 standards, not including a blank.

13.2 The ICAL must be verified with a second source standard (ICV) prior to the analysis of samples.

13.3 Results of samples not bracketed by the ICAL range must be qualified on the final report. If possible, dilute the sample or distill a smaller amount and reanalyze in order to achieve a result within the calibrated range of the instrument.

13.4 The lowest calibration standard may establish the reporting limit: RL = 0.05 mg/L for waters and 2.5 mg/Kg for soils (as received basis). The RL must be greater than or equal to the detection limit.

13.5 Samples must be quantitated from the initial calibration curve and may not be quantitated from any instrument CCV.

Initial Calibration Verification (ICV)
In addition to the method requirements, the following ICV criteria also apply:

13.6 Must be a second source standard from the ICAL standards or from a different manufacturer lot number.

13.7 Must be traceable to NIST when available.

13.8 Must be analyzed when an ICAL is not performed on the day of analysis, prior to sample analysis.

13.9 Initial Calibration Blank (ICB): (0.2% H_2SO_4) Analyzed immediately after the ICV. Acceptance limits are ± RL.

Continuing Calibration Verification (CCV)
In addition to the method requirements, the following CCV criteria also apply:
May be analyzed at the beginning of the batch to check the CCV recovery.

13.10 Must be analyzed after every 10 samples and at the end of each analytical batch.

13.11 If the CCV results obtained are outside the acceptance criteria, corrective actions must be performed. If routine corrective actions fail to produce an acceptable *second consecutive (immediate)* CCV, then either the lab has to demonstrate performance after corrective action with two consecutive successful CCVs, or a new ICAL must be performed. If the instrument has not demonstrated acceptable performance, sample analyses cannot continue until a new ICAL is established and verified with an ICV. However, sample data associated with an unacceptable CCV may be reported as qualified data under the following special conditions:

13.11.1 When the acceptance criteria for CCV are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification must be reanalyzed after a new ICAL has been established, evaluated and accepted.

13.11.2 When the acceptance criteria for the CCV are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification must be reanalyzed after a new ICAL is established and verified with an ICV.

13.11.3 When the acceptance criteria for the CCV are exceeded and it is not possible to reanalyze the sample due to limited sample quantity AND a new sample cannot be obtained by the laboratory, the data may be reported with the appropriate data qualifiers if the client has been contacted and agrees, in writing, to accept the qualified data.

13.12 Continuing Calibration Blank (CCB): (0.2% H_2SO_4) Analyze immediately after the CCV. Acceptance limits are ± RL.

13.13 Records: Initial and Continuing Calibration Records will contain, at a minimum, the following:

1. Calibration date
2. Test method
3. Instrument
4. Analysis date
5. Each analyte name
6. Analyst's initials or signature
7. Standard Concentration (appropriate units) and number of standards
8. Response (appropriate units)
9. Calibration curve or response factor
10. Evaluation of and Statistics for ICAL curve fit in order to judge calibration curve acceptance
11. Evaluation of and Acceptance Limits for ICV analysis in order to judge calibration curve acceptance
12. Evaluation of and Acceptance Limits for CCV analysis in order to judge continuing calibration acceptance
13. Calibration Standards and Reagent Solutions IDs

Calibration Acceptance Summary:

Table 2 Calibration Requirements

QCI	Frequency	Standards	Control Limits	Corrective Action
ICAL	Daily or as needed,	Minimum 5 standards, see Table 1 for concentrations	r = 0.995	Correct problem then repeat initial calibration
ICV	After each new ICAL And at the beginning of each analytical run.	1.0 mg/L	± 10% of true value	Correct problem then repeat initial calibration
ICB	After each ICV	0.2% H_2SO_4	± reporting limit	Correct problem then repeat initial calibration
CCV	Beginning (optional), every 10 samples, and end of the batch	2.5 mg/L	± 10% of true value	Correct problem then repeat CCV or repeat initial calibration
CCB	After each CCV	0.2% H_2SO_4	± reporting limit	Correct problem then repeat CCV or repeat initial calibration

Support Equipment:
Autopipettes - Check autopipette to ensure standardization is within control limits (see SOP 1040 General Laboratory Practices for Pipette Calibration).

Balances - Be sure the balance is checked prior to use and performance criteria are met (see SOP 1040 General Laboratory Practices for Calibration of Balances).

14.0 Procedure (14.1 through 14.11 for the Phenate method)

14.1 **Instrument Start Up**

14.1.1 Turn on the power to all modules by turning on the power strip and allow the autosampler to perform its startup routine. Wait until the autosampler stops with the probe above the wash bath.

14.1.2 If necessary, install the manifold on the channel you want to run. (See Appendix E.)

14.1.3 Make all the injection fluidic connections.

14.1.4 Make all the cell fluidic connections.

14.1.5 Set all pump tubes on the pump. (Varies method to method.)

14.1.6 Run reagent water through all the lines to make sure there are no leaks.

14.1.7 If there are no leaks, put actual reagents in line.

14.1.8 **ALWAYS** start pumping Buffer Solution FIRST.

14.1.9 Pour the calibration Standards into Standard vials, and place into the autosampler Standards Rack.

14.1.10 Pour the Samples into test tubes and place into the Sample Rack.

14.1.11 If necessary, insert the interference filter into the upper slot in the detector.

14.1.12 Turn on the Heater by increasing the Set Point to 60°C. Press the Set Point Key (Left of the arrow keys) until the display shows the letters SP for Set Point. Press the big arrow keys for setting the temperature, press the ENTER key to save the information. Press the Set Point key once. The display will show the current temperature in Celsius. The red light will be lit when the heater is ON.

14.2 **Software Set up**

14.2.1 Double click on the **Omnion FIA** icon. Then click **OK**. The autosampler probe should go into the wash bath and the dilutor activity may be heard, the injection valves may turn to the inject state if they were not already there.

14.2.2 Log In with your user name and password. [User name: demo with no password will also get you in]. The Main Menu should appear.

14.2.3 From the Main Menu click on the Instrument button labeled **FIA Instrument 1**. Each valve allocated to this instrument will be cycled in turn from Load to Inject and back to Inject again. Omnion will automatically open the last Method and Tray that was used. (The names will appear at the top in the title bar.).

14.3 **Open the Ammonia Method (To Create a Method see Appendix B)**

14.3.1 Click on the **Method** button, or from the Main Menu click on **File**, then **Open Method.** This will open the Open Method Dialog window.

14.3.2 Double click on the method file you want to open: **Ammonia met.** or click once then click on **OK.**

14.3.3 From the Main Menu click on the Instrument button labeled **FIA Instrument 1.** Each valve allocated to this instrument will be cycled in turn from Load to Inject and back to Inject again. Omnion will automatically open the last Method and Tray that was used. (The names will appear at the top in the title bar.)

14.4 **Opening and Editing the TRAY**

14.4.1 Click on the **Tray** button or from the Main Menu, click on **File,** then **Open Tray.**

14.4.2 Click twice on the name of the tray you wish to open. (**Ammonia tray**) The First rows of the tray spreadsheet are blue. This denotes that these samples are actually Calibration Standards. This is from the method and will not change. The Sample **Type** is **Cal Std.**

And the **Level** is NEVER 0; it will be a number from 1 to 14 (This will match the level in the Analyte Table in the method.) The Cup Number (**Cup #**) refers to the cup number in the Standard Rack and will be a number between 1 and 14.

14.4.3 Edit the **Sample ID**. Using the mouse, move the pointer to the cell and click once; type in the new ID. In the Sample Rack Loading Aid (Top Left) the sample cup you are editing will be in green.)

14.4.4 The Cup Numbers (**Cup #**) for sample will can be anything from 1 to 90.

14.4.5 The **Level** column is **0** for all samples of **Sample Type Unknown**

14.4.6 The **Reps** for samples and standards is **1 or 2**.

14.4.7 To Schedule manual QC See Appendix C (DQM Plan)

14.5 Click on the **Run Tray** button. (Or from the Main Menu, click on **Tray** then **Run Tray**.)

14.5.1 Leave the Method and Tray boxes empty to use the ones that are active (open).

14.5.2 In the Data File box enter the name for your run, (i.e. 981006Ca. YYMMDD C (for Calibration) and a (for number of the run... a b c...).) The extension *.fdt (FIA Data) is used by default.

14.5.3 The Autosampler Position refers to the autosampler sample rack position: 1, 2 or 3.

14.5.4 **Skip Recalibration Block** Box can be checked when you want to run a tray that contains Cal standards and samples but you want only want to run samples

14.5.5 While the tray is processing, you can view the peaks and runtime report.

14.5.6 If the baseline does not appear on the screen change Display options.

14.5.7 Click **Method**, then **Display Options**

14.5.8 Specify the channel: 1.

14.5.9 Specify the voltage scale on the Y-axis (i.e. -1 to 3).

14.5.10 Click **OK**.

14.6 Analyze samples for Ammonia. Click on the **RUN** button. The tray will start running. After the pump is allowed to pump at a normal speed for the period specified it the method, pump timing, the autosampler will move to the first calibration standard. The Standards and samples are done in Row order (on the computer screen), no matter where they are located on the autosampler tray.

14.7 While running you will see a **STOP** sign on the tool bar. To suspend the tray click on the STOP sign. The Tray will pause and the computer gives you the choices of aborting or resuming the tray. At the end of the tray, after the last sample ID entered is reported, the sample probe should return to the rinse position and the **STOP** button will turn back into the **RUN** button.

14.8 If baseline drifts or other problems with precision arise, clean the manifold using the following procedure. Place all reagent lines in reagent water for 5 min. Place all reagent lines in 1 M Hydrochloric Acid (1 volume concentrated HCl added to 11 volumes of reagent water) and pump for 10 min. Rinse with reagent water for 30 min.

14.9 System Shutdown Procedure

14.9.1 Remove the reagent transmission lines and place into the rinse solution and pump for 5 minutes at standard speed.

14.9.2. Place the lines into reagent water and allow the system to rinse 5 to 10 minutes at standard speed.

14.9.3 Remove the lines from the reagent water and allow all liquid to be pumped out of the manifold.
14.9.4 Turn off the pump and release the pump tube cartridges tension. Press the tube cartridges holders on the sides of the Isomatec pump.
14.9.5 Turn off the Heater by Reducing the Set Point to a temperature lower than room temperature. (e.g. 15°C).
14.9.6 Close all files
14.9.7 Switch off the master power strip

14.10 Documentation requirements. Record the following information in the appropriate logbook or data file. Include any deviations from this procedure.

14.10.1 Analyst initials, date [and time if required by the specific project or QAPP] of analysis, sample number or ID, initial sample volume or weight processed, final digestate volume, calibration standard sample or solution identifier, QC sample or solution identifier, reagent solutions identifiers, any dilution information, [beginning and ending times of analytical steps if required by the specific project or QAPP], data file name or batch ID, instrument method name, visual observations, and any other information as deemed necessary

14.10.2 Print out a copy of the calibration curve used and datafile (run sequence).

14.11 Routine Maintenance – Record all maintenance in the logbook. See Appendix F

14.12 Titrimetric Method: Fill burette with reagent upto the Zero mark (meniscus at 0). Place 50 mL. of the blank distillate in a beaker. Add titrant dropwise while swirling the beaker. Record amount of titrant used when color turns and remains lavender for 30 seconds. Repeat procedure for samples, LCS, MS/MSD.

15.0 Data Reduction, Calculations and Loading

15.1 The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

15.2 Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard must be diluted and reanalyzed.

15.3 Aqueous Samples: The concentration readout for aqueous sample distillates is mg Ammonia as Nitrogen/L. It does not need further data reduction unless the initial sample volume was less than 50 mL. If less than 50 mL sample was distilled, calculate the concentration as follows:
Concentration in mg Ammonia /L = readout * (50 ml/ initial sample volume analyzed in mL)

15.4 For sample results greater than the highest calibration standard, dilute the sample in a 10 mL centrifuge tube using Reagent 5.

15.5 Pipette in the appropriate volume of sample into the tube, dilute to the mark with Reagent 5. Record the volume of distillate used for analysis. Dilution Factor = (10 mL/ distillate volume analyzed in mL). Use the dilution factor and calculate the concentration in aqueous samples as follows:

Concentration in mg Ammonia /L = readout * (10 ml/ distillate volume analyzed in mL)

15.6 Soil Samples: The concentration readout for soil samples must be multiplied by the following factor: Factor = (50 / sample weight in g). Calculate the concentration in soil samples as follows:
Concentration in mg Ammonia /Kg = readout * (50/ sample weight in g)

For soil sample results greater than the highest calibration standard, follow the dilution procedure in section 15.4

15.7 Soil samples reported on a dry weight basis: The concentration is divided by the decimal equivalent of the percent residue of the soil at 105^0C.

15.8 Report results in mg Ammonia /L, or mg Ammonia /Kg.

15.9 The procedure for uploading data into the LIMS system is detailed in SOP 1400 LIMS.

15.10 For the Titrimetric Method,

Mg NH3-N/L = (A-B) x 280/mL. sample

Where, A: Volume of H_2So_4 required for sample (mL)
B: Volume of H_2So_4 required for blank (mL)

16.0 Method Performance

Demonstration of Capability (DOC)

Note: Each analyst must demonstrate the ability to generate acceptable results with this method.

All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

A quality control (QC) reference concentrate is required containing Ammonia at a concentration of 1-4 times the reporting limit. The QC reference sample is made using stock standards prepared independently from those used for calibration. For the Phenate Method, distillation is required. For the Titrimetric method, disrtillation is not required.

For each analyte, calculate the mean recovery (X), standard deviation (s), relative standard deviation (RSD), and the average % Recovery (%R). Compare X and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. Note: RSD must be equal to or less than 20% and the %R must be within 100 ± 20%. These limits are taken from established in-house criteria If RSD and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If RSD or %R falls outside the range for accuracy and precision, then the system performance is unacceptable for that analyte and corrective action must be taken.

Comparison to Reference Method Data
There are no stated reference method criteria for ICV, LCS, Duplicate Sample or MS/MSD recoveries in Method 4500-NH_3.

In-House Control Limits
Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data is compared to the specifications of the reference method. If the in-house limits are within the specifications of the reference method, the control limits are updated in LIMS. If the in-house limits are not within specifications, an investigation is performed to determine the cause(s) of the problem and a corrective action is completed. The analysis may continue until enough data points are collected to regenerate new control limits. Any QC data generated outside of reference method limits during that time frame is flagged.

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

17.0 Pollution Prevention

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the laboratory waste disposal program and applicable waste disposal regulations. With the consent of the client, the samples may be returned to their origin for treatment.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory staff are required to protect the laboratory's and our clients' business information when disposing of recycling or waste from the facility.

18.0 Data Assessment and Criteria for Quality Control Measures

The laboratory maintains records to document the quality of data that is generated. Ongoing quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. The data review is conducted according to SOP 1250 Data Review.

Method Blank (MB)
If the blank exceeds the RL (the lowest calibration standard), the source of contamination must be investigated and corrective actions taken.

Affected samples must be reprocessed and reanalyzed or Data must be appropriately qualified if:

1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the SOP or by regulation, AND is greater than 1/10 of the amount measured in any sample.

2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

Laboratory Control Sample (LCS)
The results of the individual batch LCS are calculated in percent recovery (%R) and compared to established acceptance criteria (in-house limits). LCS %R limits are 100 ± 20%. If the LCS is outside the acceptance criteria, the analytical system is "out-of-control". Any affected samples associated with an out of control LCS must be reprocessed and reanalyzed or the results reported with appropriate data qualifiers.

Matrix Spikes
The results from MS/MSD are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). See the STAT QAM, Section 5.4 for the calculation for RPD. Results are compared to established acceptance criteria (in-house limits). For aqueous samples, MS/MSD %R limits are 100 ± 25% and RPD limits are 20%. For soil samples, MS/MSD %R limits are 100 ± 25% and RPD limits are 20%. For matrix spike results outside established criteria corrective action must be documented, or the data for the spiked sample is reported with appropriate data qualifying codes.

Duplicates

The results from laboratory Duplicates are designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). See the STAT QAM, Section 5.4 for the calculation for RPD. Results are compared to established acceptance criteria (in-house limits). RPD limits are 20%. For duplicates results outside established criteria corrective action must be documented, or the data for the duplicate sample is reported with appropriate data qualifying codes.

19.0 Corrective Actions for Out-of-Control Data

The process for handling corrective actions is found in SOP 230 Corrective Action.

If the CCV, MB, LCS/LCSD, MS/MSD, or lab duplicate recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the samples is suspect and is only reported for regulatory compliance purposes with the appropriate corrective action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. Final data results must be qualified in the client report for reported results not meeting the laboratory-defined criteria.

1) Review standards preparation logbooks. Check all calculations and ensure dilution factors are properly recorded.

2) Re-prepare the suspected standard or QC sample to identify possible preparation errors of the standard or QC sample.

3) Re-Analyze the samples when the CCV or LCS is not within acceptable limits.

4) Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

20.0 Contingencies for Handling Out-of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discuss options with the client. Reanalysis or reporting the data with qualification is alternatives. Out-of-control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.

The process for handling unacceptable and out of control data is found in section 11 of the Laboratory QAM. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21.0 Waste Management

The STAT Analysis Corporation SOP 1130 Waste Disposal identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded accordance with SOP 1130 Waste Disposal.

22.0 References

22.1 Method 4500-NH_3 B, H, and C. U.S. EPA, Standard Methods for the Examination of Water and Wastewater (20^{th} Edition).
22.2 Determination of Total Recoverable Ammonia by Flow Injection Analysis. QuikChem Method 10-107-06-1-B.
22.3 STAT Analysis Corporation Quality Assurance Manual
22.4 STAT SOP 003 Chemical Hygiene Plan
22.5 STAT SOP 230 Corrective Actions
22.1 STAT SOP 1000 Control and Use of Laboratory Notebooks
22.2 STAT SOP 1010 Standard and Reagent Preparation
22.3 STAT SOP 1020 Glassware Cleaning
22.4 STAT SOP 1040 General Laboratory Procedures
22.5 STAT SOP 1130 Waste Disposal
22.6 STAT SOP 1210 Method Detection Limits (MDL's)
22.7 STAT SOP 1230 Training
22.8 STAT SOP 1250 Data Review
22.6 STAT SOP 1400 LIMS
22.7 STAT SOP 3250 Ammonia Distillation by EPA 4500-NH_3 B.
22.8 QuikChem 8000 Automated Ion Analyzer Omnion FIA Software.
22.9 QuikChem 8000 Automated Ion Analyzer Continuum Series "Flow Injection Analyzer" Hardware Installation and System Operation.

23.0 Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

23.1	Appendix A	Troubleshooting
23.2	Appendix B	Creating a Method
23.3	Appendix C	DQM Plan
23.4	Appendix D	Manifold Diagram
23.5	Appendix E	Manifold Installation/ Removal
23.6	Appendix F	Maintenance Schedule

Appendix A – Trouble Shooting

Keep all modules clean and dry at all times.

Keep in Stock: Pump tubes orange
White
Red
Red
Gray
Green
Teflon tubing: 0.8 mm id Manifold
0.5 mm id Low Flow Manifold
0.6 mm id Restriction Coil
O-rings
Transmission Tubing

Routine Maintenance (See Appendix F for the Maintenance Schedule)

Pump

- After each day rinse the cartridges in reagent water to wash any spills. Clean pump surfaces, except rollers, with a wet cloth. Dry all surfaces.
- If rusty clean rollers with steel wool. Apply silicon spray on a lint free cloth and holding it to the moving rollers to apply a light coat of silicon.
- Check for wear, cracks or acid damage on the cartridges ad holders.
- Replace pump tubes that start to show signs of wear
- If the pump tubes burst, clean all cartridges and holders, as well as, the pump, immediately.
- All surfaces should be kept clean. Use a damp cloth to clean the module surfaces. Dry surfaces thoroughly.

Valve Modules

- Keep the instrument clean and dry at all times.
- Replace o-rings once per month
- When changing the o-rings, clean the valve ports with cotton swab and reagent water. This will help remove any dirt or precipitate that may prevent a good seal. If a leak persists, replace the new o-rings and make sure the connector itself does not have precipitate on the thread.
- All surfaces should be kept clean. Use a damp cloth to clean the module surfaces. Dry all surfaces thoroughly.
- The internal sample loop valve will give thousands of injections without trouble. The rotor seal wears with use and is the only part that needs routine replacement.

Detector Modules and Flow Cell

- If a flow cell appears to leak remove it immediately to keep all liquid form the electronics inside the detector head.

Instrument Troubleshooting

For problems with the instrument see the QuikChem 8000 Automated Ion Analyzer Continuum Series "Flow Injection Analyzer" Hardware Installation and System Operation Manual under the Troubleshooting Section.

Appendix B – Creating A Method

1.1 Click on the **Method** button, or from the Main Menu click on **File**, then **New Method**.
1.2 Then Method will open and you will see the Analyte Table
1.3 Click on Analyte Name of channel and press backspace to delete. Enter the new analyte name. If an analyte name is not present, the system will ignore that channel.
1.4 Fill out the analyte Table.

Channel	1
Analyte Name	Ammonia
Concentration	mg/L
Level 1	5
Level 2	2.5
Level 3	1
Level 4	0.5
Level 5	0.1
Level 6	0.05
Level 7	0.01
Level 8	0

Calibration Rep Handling	Average
Calibration Fit Type	1st Order Polynomial
Force Through Zero	No
Weighting Method	None
Concentration Scaling	None
Chemistry	Direct
Injection to Start Peak	41.8 s
Peak Base Width	27.8 s
% Width Tolerance	100%
Threshold	10000

1.5 From the Main Menu, click on **Method**, then **Valve Timing**
1.6 Enter the **Method Cycle Period** 60s
1.7 Sample Reaches the first valve 18s (For Standard Pump Sample Assembly) (travel time from sample probe to port 6 usually 24 s if dilutor enter 28s)
1.8 **Load Period** 15s
Load Time 0s
Inject Period 45s
Notice that Load Period + Injection Period = Cycle Period
1.9 Click **OK**

1.10 From the Main Menu, click on **Method**, then **Sampler Timing**.
1.11 The Sample Prep Sequence box is optional
1.12 Enter the **Minimum Probe in Wash Period** 5.0 s
1.13 Enter the **Probe in Sample Period** 24s

1.14 Click **OK.**

1.15 From the Main Menu, click on Method, then Pump Timing
1.16 Check the **Go to Standby on Idle** box X
1.17 Enter **Idle Before Standby** 180.0s
1.18 Enter **At Speed Before Analysis** 45.0s
1.19 Click **OK.**

1.20 From the Main Menu, click on **File**, then **Save Method As**
1.21 The FIA Method File Header will appear. Write a method description (analyte and sample loop size) and click **OK.** (i.e. **Ammonia Sample Loop XXX cm)**
1.22 The Method Save As dialog box will appear.
1.23 C:\ Omnion\Methods\
Change the name to **Ammonia.met**

II To Fine Tune the Method
1.0 **After you have run a data.**
1.1 Open a file from the main menu click on File, Open Data and click twice on the Data file name, i.e. 981006Ca.fdt.
1.2 Load the original method form this data by clicking on the Data menu the Load Method.
1.3 You should see the peaks on the screen.
1.4 Click on Data then Reanalyze Data. (Or click on the Reanalyze button.)

2.0 **Fine Tuning the Threshold**
2.1 Look before and after the tray to see some baseline.
2.2 From the menu click on **Method**, then **Graphical, Events, Programming...**
2.3 Click on **Threshold.**
2.4 The Status bar will prompt you to "Select Start of Baseline Section".
2.5 Set the cursor at the beginning of the baseline region and click once.
2.6 You will be prompted to "Select End of Baseline Section".
2.7 Move the mouse pointer to the end of the baseline region and click again.
2.8 Omnion calculates a Threshold value.
2.9 Click on **OK** to enter the value in the Analyte Table of your method

3.0 **Fine Tuning the Peak Base Width**
3.1 From the menu click on **Method**, then **Graphical Events Programming...**
3.2 Click on **Peak Base Width.**
3.3 You will be prompted to "Select Start of Peak"
3.4 Set the cursor at the beginning of the high standard and click once.
3.5 You will be prompted to "Select End of Peak"
3.6 Move the cursor to the end of the peak and click again.
3.7 Omnion calculates a Peak Base Width.
3.8 Click on **OK** to enter the value in the Analyte Table of your method

4.0 Save these new method parameters by clicking on **File**, then **Save Method As.**

5.0 **Viewing a Method's Calibration**

5.1 Up to 4 replicates of each Standard can be applied to a method's calibration.

5.2 If not already open, load the method form the data file by clicking on Data, Load Method.

5.3 Click on the button. (Or from the main menu click on **Method**, then **Review Analyte Calibration Curve**. The Review Analyte Calibration window will appear.

5.4 Click **Fit**, and then **Clear** to 'Clear' the Calibration Replicate Table.

5.5 Click **Exit**.

5.6 Click the **Analyze** button. The re-analysis occurs exactly as it did in the actual tray run.

5.7 Click on the **Review Calibration** button again to see the curve.

6.0 **Editing the Calibration**

6.1 Clicking twice on any of the results in the calibration replicate Table will turn it red and make it unused in the calibration.

6.2 To Use the point again, click twice on it again, and it will turn form red back to blue.

Appendix C – DQM Plan

DQM Plan

Consists of one or several DQM sets. Each Set has one or more samples. The DQM has 3 sections; the DQM set box, the DQM Sample Box, and the Channel Data box.

1.0 **Adding DQM Sets**

1.1 Click Tray, then Load DQM.

1.2 In the DQM Set Box click on the drop-down button showing the **DQM Set ID**, type in a new name. (Check Standards, Duplicates, Matrix Spikes)

1.3 For the Check Standards Set check the **Automatic** box **X**.

1.4 Click **ADD** to add the set.

NOTE: Automatic Sets never have their sample info in the tray table and the samples are loaded in the standards rack. Manual sets have their sample info in the tray table and the samples are loaded in the sample rack.

2.0 **Adding DQM Samples**

2.1 In the DQM Sample Box, click on the drop-down button showing the **DQM Sample ID** and type in a new name (ICB, ICV, CCV, CCB, Method Blank, Dup 1, Spike, Spike Dup)

2.2 Click on the **Append** button.

2.3 **Replicates** should be **2**

2.4 Click on the drop-down button showing the **Type** and select the type of sample. (Blank, Unspiked, Spiked, AbsChkStd, RelChkStd, Dup 1, Dup 2)

2.5 For the Automatic Check Samples enter the **Standard Rack Cup.**

3.0 **Editing Channel Data information.**

3.1 In the Channel Data Box, select the appropriate channel.

3.2 If you can't see the Test 1 row, click on the **Add Test** button.

3.3 The Test Explanation spells out the test that will be done for this DQM Sample and depends on what Type the DQM Sample is.

3.4 If the standard has a known concentration it needs to be entered in the **Known Conc** box along with the **Conc Units.**

3.5 Enter the **Test Limit.** 10.000% difference.

3.6 Select a Fail Action from the drop-down menu. **Recalibrate & Repeat, Alarm & message.**

3.7 Enter the Pass/ Fail Message.

NOTE: A Test Passes if test value < = Test Limit
A Test Fails if Test value > Test Limit

3.8 You can perform more then 1 test on each check sample.

4.0 **Scheduling Automatic DQM Sets**

4.1 From the Main Menu, click on **Tray**, then **Auto DQM Schedule**

4.2 Select the **Auto DQM Set** from the drop-down menu.

4.3 Check the box (es) for the frequency of the sample

4.4 Click **OK.**

5.0 **Inserting Manual DQM Sets**
5.1 Click on the **Tray Table** button.
5.2 Click on the row number before which you want to insert the check sample.
5.3 From the Main Menu, click on **Tray**, then **Manual DQM Insertion**.
5.4 Click on the **Manual DQM Set** you wish to insert.
5.5 Enter the **Sample ID**'s of the DQM samples to reflect the actual identity of the samples. (Note: The manual DQM sample rows are green.)
5.6 Renumber the Cup Numbers (**Cup #**) below the inserted Manual DQM Set by clicking and dragging all the rows and cup numbers you wish to renumber.
5.7 Click on **Tray**, then **Renumber Cups** (or **Ctrl-R**)
5.8 Enter the **Starting Number**
5.9 Enter an **Increment** of **1.**
5.10 Then click **OK.**

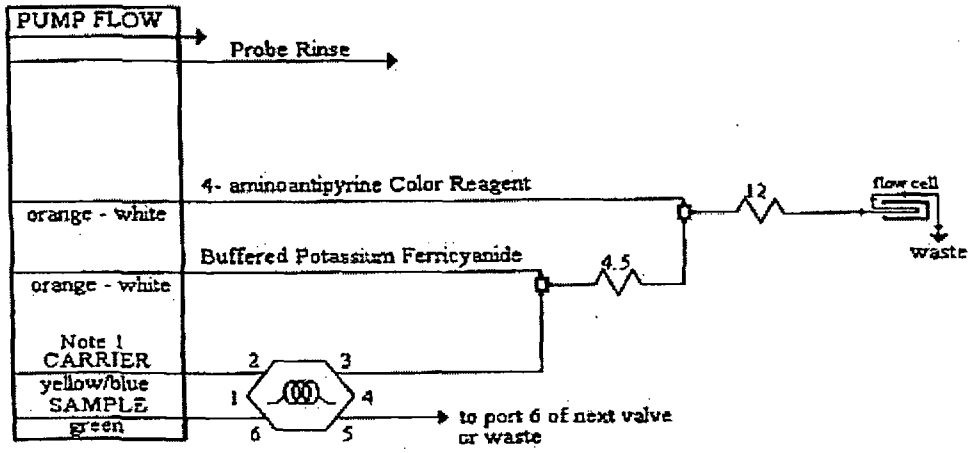
6.0 **Calibration Pass/Fail Criteria**
6.1 From the Main Menu, click on Method, then Calibration Failure Criteria
6.2 Select the appropriate channel.
6.3 Check the Minimum Correlation Coefficient (R2) Box and enter 0.995
6.4 Check the Maximum % Residual All Levels Box, and enter 10.0.
6.5 If the calibration passes, a message will be reported and the tray will continue to the nest row.
6.6 If the calibration fails, a fail message will be reported and you will be given a choice to Abort or continue the tray.

7.0 **Save the DQM Plan**
7.1 From the Main Menu, click File, then Save DQM Plan, or Save DQM Plan As... and enter the File name Ammonia.dqm. The default extension is *.dqm.

Appendix D – Manifold Diagram

17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1. PHENOLICS MANIFOLD DIAGRAM:



Sample loop = 150 cm
QC8000 sample loop = 155.5 cm

Interference Filter = 500 nm

Manifold tubing is **0.8 mm (0.032 in)** i.d. This is 5.2 uL/cm.

CARRIER is helium degassed water.

4.5 is **70.0** cm of tubing on a 4.5 cm coil support

12 is **255** cm of tubing on a 12 cm coil support

APPARATUS: Standard valve, flow cell, and detector head modules are used.

Note 1: **Carrier Line** for the AE: use a green/green pump tube.

Note 2: Transmission tubing should be replaced with 1 m of Teflon manifold tubing (0.8mm i.d.) as transmission tubing may contain leachable phenolics. Use Teflon tube connectors (Part# 50008) with PTA as line weights with pin removed.

Appendix E – Manifold Installation/Removal

Manifold Installation Procedure

1.0 Unwrap the transmission lines from around the manifold and place the manifold over the sample-processing module.

1.1 Remove the transmission lines from the union on the 650 cm side of the heating unit, insert both ends through the hole in the manifold and reseat the manifold onto the sample-processing module.

1.2 **Make the Injection Valve Connections as follows:**

1.3 Port 1 & 4 - The Sample Loop 0.8 mm i.d. length 75 cm.

1.4 Port 2 - Carrier line is connected to Port 2 -> from valve, disconnect from Fitting and connect to port 2 0.8 mm id and 30 cm long.

1.4 Port 3 - 20 cm 0.8 mm id between port 3 and the fitting on the manifold next to the label -> from valve where the carrier line was connected.

1.5 Port 5 - 15 cm 0.8 mm id. between port 5 and the waste line.

1.6 Port 6 - Sample Line. 130 cm (Varies method to method) connected to the probe on the autosampler; pump tube adapter, and 20 cm tubing connected to port 6 of valve.

2.0 **Flow Cell** Top tubing connected to waste line. Bottom line connected to the fitting in the manifold next to the label to flow cell ->.
Attach one of the lines from the heater to the union going to the flow cell.
Attach the other heater line to the pyridine-barb. acid Tee-fitting.

3.0 **Pump**

3.1 Sample Line - Autosampler to injection valve 6.

3.2 Wash Line - Reagent water to wash reservoir on the autosampler

3.3 Reagents Lines- Varies from method to method sees Ammonia diagram.

3.4 On the Ismatec Pump Cartridges the arrows point towards the System Unit with the tension lever on the left.

3.5 Place all of the reagents lines into corresponding containers or Reagent water.

3.6 Move tension levers to the maximum tension (Top Far Right Position)

3.7 Clamp down all pump tube cartridges. (Press down one side at a time.)

3.8 Move tension lever back from the top far right until it makes a clicking sound.

3.9 Set the reagent pump speed to 35.

3.10 Turn on the pump.

3.11 Depress the green button to turn the pump ON. The System Unit will take control over the pump speed. (The yellow button is the override standby button.)

3.12 Check to confirm that the probe wash reservoir is filing with rinse water.

3.13 Check for Leaks on the manifold, valve, flow cell or any of the connections.

3.14 Do NOT leave any pump tubes clamped down when the pump is shut off for more then a few minutes.

3.15 To Remove cartridges, Press the sides of the pump holder on which the cartridge is engaged.

4.0 Insert the interface filter into the detector module.

II Manifold Removal Procedure

1.0 Rinse the manifold

1.1 Detach the manifold tubing from the manifold fitting that is connected to Port 3 at the injection valve. Leave the piece of tubing attached to the injection valve.

1.2 Disconnect the carrier pump tube from Port 2 of the injection valve. Take this tubing and connect it to the manifold fitting that was connected to Port 3.

1.3 Detach output of the manifold from union on the flow cell tubing leave the union connected to the flow cell

1.4 Remove the back pressure loop, if necessary

1.5 Detach heating unit tubing from the manifold, and reconnect to the union underneath the manifold (650 cm side.)

1.6 Remove all manifold pump tubes from cartridges.

1.7 Remove the interface filter from the detector module

1.8 Remove the sample loop from Port 1 & 4 valve

1.9 Remove manifold from the Sample Processing Module (Channel)

1.10 Carefully wrap the transmission lines around the manifold and store it in the plastic bubble bag.

Appendix F – Maintenance Schedule

All listed maintenance is performed as needed.
Following is a checklist of items along with the maintenance that may need to be performed. It is the instrument analyst's responsibility to check the condition of the instrument daily and perform any necessary maintenance to insure the instrument is operating correctly.

AutoSampler	Clean Surfaces
AutoDilutor	Clean Surfaces Prime dilutor with reagent water after using any other diluent
Pump	Clean Surfaces Rinse cartridges
Detector	Dry and clean all Surfaces
System Unit	Keep Dry and Clean
Injection Valves	Clean Ports and valve connections
Autosampler Pump	Clean rods/ moving parts Replace pump tubes as needed Clean pump tube adapters
Manifold	Clean union and tee as needed
Injection valves	Replace o-rings
Manifold	Replace o-rings
Manifold	Check all tubing and replace as needed
Flow Cells	Check and replace flares and o-rings as needed
Computer	Clean hard drive

STAT Analysis Corporation:

STANDARD OPERATING PROCEDURE 4510

METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY (EPA METHOD 6020 AND EPA METHOD IO-3.5)

Revision 05

Effective Date: May 16, 2005

Author: Dennis Jachim, Bruce Anderson

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Table of Contents

1.0 IDENTIFICATION OF TEST METHOD

SOP Title: Metals Analysis by Inductively Coupled Plasma - Mass Spectrometry (EPA Method 6020 and EPA Method IO-3.5) is abbreviated as ICP-MS in the laboratory records.

2.0 APPLICABLE MATRICES

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of µg/L and sub-µg/L concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for aqueous, solids, and air filters for which total (acid-leachable) elements are required.

3.0 DETECTION LIMITS

The lab follows the procedure found in 40CFR Part 136B to determine the MDL for each matrix type on an annual basis. See the STAT Analysis SOP 1210 Method Detection Limits for the MDL procedure, frequency, and acceptance criteria. The MDLs measured by the lab and all supporting documentation are in the laboratory QA files for review.

The laboratory determined method detection limit (MDL) must always be less than the reporting limit (RL). The RLs will usually range from three to ten times the laboratory measured MDLs but this relationship may vary dependent on dilution, reduced sample size to avoid saturation of the detector, matrix interferences, moisture adjustments (in solid samples), or method-specified requirements. Attachment 1 contains the current MDLs and reporting limits (RLs) for soils, waters and air filters.

4.0 SCOPE AND APPLICATION

4.1 Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-µg/L concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, air filters, wipes, and other solid wastes for which total (acid-leachable) elements are required.

4.2 ICP-MS has been historically applied to the determination of over 60 elements in various matrices. Analytes determined at this laboratory for this method are listed in Attachment 1 (along with the masses used for determination). If Method 6020 is used to determine any analyte not listed in Attachment 1, it is the responsibility of the laboratory to demonstrate the accuracy and precision of this method in the material to be analyzed. The analyst is always

required to monitor potential sources of interferences and take appropriate action to ensure data of known quality (see Section 14.4 Qualitative Analysis).

4.3 This method is restricted to use by or under the supervision of analysts experienced in the use of ICP-MS and knowledgeable in the recognition and correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 SUMMARY OF TEST METHOD

5.1 Samples that require total "acid-leachable" values must be digested using appropriate sample preparation methods prior to analysis.

5.2 EPA methods 6020 and IO-3.5 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix. An appropriate internal standard is required for each analyte determined by ICP-MS.

5.3 Method Modifications from Reference – This SOP is based on EPA Method 6020 and incorporates IO-3.5 for TSP/PM10 high-volume filters except that: 1) polypropylene volumetric flasks are used since analysis for boron is occasionally required, 2) the instrument resolution is set for 1 amu peak width at 10% peak height according to 6020 but is less stringent than IO-3.5, 3) samples which fail high or low for one or more internal standards are diluted as needed to bring within range for the internal standard, 4) A matrix spike and matrix spike duplicate are digested and analyzed, since a duplicate sample is not typically analyzed, 5) Dilution test may be performed on aqueous matrices. Non-aqueous matrices are already diluted before analysis 6) reagent water must be at least Type II (ASTM) for all standards, solutions, and sample preparation according to 6020 but is less stringent than IO-3.5, 7) internal standards and tuning solutions cover the entire mass range of analysis, but the elements used vary slightly from IO-3.5, 8) the MDLs are determined annually according to the requirements described in SOP 1210 Method Detection Limits 9) the ICB/CCB acceptance limit is the lower reporting limit and not the MDL per IO-3.5, since we do not report down to the MDL. 10) Germanium instead of yttrium is used as an internal standard, since yttrium is found in environmental samples.

6.0 DEFINITIONS

The STAT Analysis Quality Assurance Manual Section 19.0 contains all the definitions of standard terms used in SOPs.

7.0 INTERFERENCES

7.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

7.2 Isobaric molecular and doubly charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar\ ^{37}Cl$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl$ signal): corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = (m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal), (where the final term adjusts for any selenium contribution at 77 m/z).

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes. Similarly, corrected cadmium signal (using natural isotopes abundances for coefficient approximations) = (m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal), (where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114).

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are **ONLY** illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision. The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This

type of correction has been reported for oxide-ion corrections using ThO^+ /Th^+ for the determination of rare earth elements. The use of aerosol, desolvation, and/or mixed plasmas has been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

7.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of an internal standard is less than 30 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

7.4 Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially, deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interference. Whenever an unusually concentrated sample is encountered, the sample after it may need to be reanalyzed to check for cross contamination.

8.0 SAFETY

Proper personal protective equipment including safety glasses, gloves and a lab coat are required during different parts of this method. Other safety precautions must be conducted in accordance with the Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of Material Safety Data Sheets (MSDS) for all reagents/chemical involved in this method is available to all personnel

Stock metal standards and acid solutions (strong oxidizers) may pose potential health risks. Extreme care should be utilized when handling these solutions.

9.0 EQUIPMENT AND SUPPLIES

The following apparatus is recommended for performing this procedure. Equivalent items can be used, if with their use, the analytical and QA/QC requirements in this SOP can be met.

9.1 Inductively coupled plasma-mass spectrometer: Aglient 7500i or equivalent.

9.1.1 A system capable of providing resolution, better than or equal to 1.0 amu at 10% peak height is required. The system must have a mass range from at least 6 to 240 amu and

a data system that allows corrections for isobaric interferences and the application of the internal standard technique. Use of a mass-flow controller for the nebulizer argon and a peristaltic pump for the sample solution are recommended.

9.1.2 Chiller - Neslab CT 100 or equivilent

9.1.3 Argon gas supply – high purity grade (99.99%).

9.1.4 Data system - An Agilent Kayak computer system using 3365 Chemstation Version C.00.01 to interpret spectral data is attached to the ICP-MS.

9.1.5 Autosampler – Cetac ASX-450 or equivalent.

9.2 Autopipettes – 0.01 to 0.10 mL, 0.1 to 1.0 mL, 1.0 to 5.0 mL.

9.3 Dispensing pipettes – 1 to 10 mL

9.4 Polypropylene Volumetric Flasks, - 50 mL, 100 mL, 250 mL, and 500 mL with screw caps. See section 14.8 for the procedure of checking the flask volume.

9.5 Plastic Tubes – 14 mL culture tubes and 50-mL graduated with screw caps.

9.6 Plastic bottles – 250 mL, 500 mL and one liter with Teflon screw caps

10.0 REAGENTS AND STANDARDS

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see SOP 230 Corrective Action).

Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagents Receipt and Preparation. All standards solutions and QC solutions are stored at room temperature in plastic bottles.

10.1 Analytical reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

10.2 Reagent water - All references to water in this SOP refers to Type II reagent water (in-house system).

10.3 Calibration Stock Standard - Standard solutions can be prepared from pure standard materials or purchased as certified solutions. The routine laboratory practice is to purchase these standards from approved vendors. These stock standard solutions are purchased or prepared from ultra-high purity grade chemicals or metals (99.99 or greater purity) for most elements. The stock standards for individual elements are usually at concentrations of 1000 or 10,000 mg/L in solution. Custom blend stock standard solutions may also be purchased and used. Stock standard solutions and QC solutions are stored at room temperature in their original containers.

10.3.1 Secondary Calibration Stock Standard – These solutions are made using the individual Stock Standard solutions. Three secondary solutions are prepared by combining and diluting the individual stock standard solutions to levels in the linear range for the instrument in a matrix consisting of 2% (v/v) HNO_3 in reagent water. When preparing the secondary stock standards, each stock solution must be initially verified to determine correct preparation and/or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. The following amounts of each Stock Standard solution are pipetted into a 250 mL volumetric flask. The solution is brought to volume with 2% nitric acid (HNO_3). The final concentration of each component is listed in Table 1. These Secondary Stock Standard solutions are the source solutions to prepare the calibration standards in Section 10.4. Fresh mixed standards must be prepared as needed with the realization that concentrations can change on aging. Calibration standards must be initially verified using a quality control standard and monitored weekly for stability.

Table 1 Secondary Calibration Stock Standard Solutions

Secondary Stock Solution	Elements	Conc. Stock (mg/L)	Vol. Stock (mL)	Final Volume (mL)	Final Conc. (mg/L)
A	Al, As, B, Ba Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Tl, V, Zn	1000 each	2.5 mL each	250	10
	Sb, Mo, Sn, Ti		1.25 mL each		5
B	Ag	1000 each	2.5 mL each	250	10
C	Na, Ca, Mg, K, Fe	10,000 each	2.5 mL each	250	100

10.4 Calibration standards - Four calibration standards are prepared. Calibration standards in current use: 1, 10, 100, and 200 mg/L (for all elements except Ca, Fe, K, Mg, Mo, Na, Sb, Sn , and Ti (see Table 2). The following amounts of Secondary Stock Standard Solutions A, B, and C are pipetted into a 50 mL volumetric flask. Each calibration standard solution is brought to volume with 2% nitric acid (HNO_3). The final concentration of each component is listed in Table 2. Level 1 Calibration standard can be prepared by taking 5 mL of level 2 and diluting to 50 mL.

Table 2 Calibration Standards

Level	Volume (µL) Secondary Stock A and B	Volume (µL) Secondary Stock C	Final Volume (mL)	Final Conc. Trace Elements * (µg/L)	Final Conc. of Sb, Mo, Sn, & Ti (µg/L)	Final Conc. Major Elements (µg/L) Na, Ca, Mg, K, Fe
1	5mL of level 2 standard		50	1	0.5	10
2	50	50	50	10	5	100
3	500	500	50	100	50	1000
4	1000	5000	50	200	100	10000

* Except Sb, Mo, Sn, and Ti.

10.5 ICP-MS Internal Standard Stock Solutions: A solution containing 100 µg/mL ^{6}Li, ^{45}Sc, ^{159}Tb, ^{89}Y, ^{115}In and ^{209}Bi. Single element Germanium Standard 1000 mg/L. These solutions are purchased commercially.

10.5.1. Working Internal Standard Solution (1 mg/L): Pipette 1.0 mL of the mixed ICP-MS Internal Standard Stock Solution and 0.10 mL of the Germanium Standard into a 100 mL volumetric flask and bring to volume with 2% nitric acid (HNO_3). The working internal standard solution is added to a reservoir and will be added on-line to each standard, QC sample, and test sample at the time of analysis using a second channel of the peristaltic pump and an appropriate mixing manifold. This will deliver approximately 40 mg/L at the instrument for every sample/standard analyzed. Generally, an internal standard should be no more than 50 amu removed from the analyte.

10.6 ICP-MS Tuning Solution Stock 10 mg/L: Commercially purchased. This solution contains 10 mg/L each of Ce, Tl, Li and Y.

10.6.1. Working ICP-MS Tuning Solution 10 µg/L: Pipette 0.25 mL of the ICP-MS Tuning Stock Solution into a 250 mL volumetric flask and bring to volume with 2% nitric acid (HNO_3).

10.7 Stock 6020 Tune Check Solution: A solution containing elements representing all of the mass regions of interest (10 mg/L each of Li, Co, In, and Tl) must be prepared to verify that the resolution and mass calibration of the instrument are within the required specifications. This solution is also used to verify that the instrument has reached thermal stability. Purchased commercially.

10.7.1. Working 6020 Tune Check Solution 100 µg/L: Pipette 1.0 mL of the Stock 6020 Tune Check Solution into a 100 mL volumetric flask and bring to volume with 2% nitric acid (HNO_3).

10.8 Stock P/A Factor Tuning Solution (10 mg/L): Using multi-element Standards prepare a 10 mg/L Standard in 2% Nitric Acid.

10.8.1. Working P/A Factor Tuning solution 100 µg/L: Pipette 1.0 mL of the Stock 6020 Tune Check Solution into a 100 mL volumetric flask and bring to volume with 2% nitric acid (HNO_3).

NOTE: Working Calibration Standards 1, 2, 3, and 4 may also be used to determine P/A Factors.

10.9 ICP-MS Interference Check Solutions A and AB: Purchased commercially. See Attachment 2 for concentrations.

Working ICS Solutions:

10.9.1. ICS-A: Pipette 20.0 mL of ICP-MS Interference Check Solution A and 2 mL of concentrated Nitric Acid into a 100 mL volumetric flask and bring to volume with reagent water. ICS solution A must be prepared fresh weekly.

10.9.2. ICS-AB: Pipette 20.0 mL of ICP-MS Interference Check Solution AB and 2 mL of concentrated Nitric Acid into a 100 mL volumetric flask and bring to volume with reagent water. ICS solution AB must be prepared fresh weekly. The analyst should be aware that the solution might precipitate silver more quickly.

10.10 Laboratory Control Sample/Matrix Spike Stock - The LCS/matrix spike is prepared from individually purchased elements or custom blend stock standards. The Calibration Verification Stock Standards or Independent Stock Standards may be used to prepare the solution. The LCS/Matrix Spike Stock is prepared according to Table 3 below. Fill a 1000 mL volumetric flask with 400 mL of 2% v/v nitric acid. Pipette the appropriate volume of each analyte into the flask and bring to volume with 2% v/v nitric acid. This solution has a shelf life of six months.

Table 3 Laboratory Control Sample/Matrix Spike Stock

Elements	Conc. Stock (mg/L)	Vol. Stock (mL)	Final Volume (mg/L)	Final Conc. (mg/L)
Al, As, B, Ba Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Tl, V, Zn	1000 each	25 mL each	1000	25
Sb, Mo, Sn, Ti	1000 each	12.5 mL ea.		12.5
Ag	1000 each	10 mL each		10
Na, Ca, Mg, K, Fe	10,000 each	10 mL each		100

Spike with one milliliter of this stock solution per 50 ml digestate when preparing the LCS, LCSD, MS, MSD. For the Post Digestion Spike (PDS) samples, spike in the ratio of 1 mL per 50 mL digestate.

10.10.1 If ambient air filters are to be analyzed for Barium or Zinc, additional spike will need to be added. The additional spike should have a concentration of 24 mg/L of Barium and 0.5 mg/L of Zinc. Spike with 1 mL of the Barium and Zinc spike. To prepare this solution, add 100 mL of 2% HNO_3 to a 250 mL volumetric flask.

Pipette 6 mL of 1000 mg/L Ba standard and 0.125 mL of 1000 g/mL Zn standard. Dilute to volume.

10.11 Calibration Verification Stock Standard Solutions (ICV/CCV) – The Calibration Verification Stock Standard Solutions A and B are prepared from 1000mg/L single element standards. These solutions are prepared by combining and diluting individual stock standard to levels in the linear range for the instrument in a matrix consisting of 2 percent (v/v) HNO_3 in reagent water. Prior to preparing the calibration verification stock standards, each stock solution must be analyzed to determine correct preparation and/or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. The following amounts of each Stock Standard solution are pipetted into a 250 mL volumetric flask. The solution is brought to volume with 2 percent (v/v) HNO_3 in reagent water. The final concentration of each component is listed in Table 4. These Calibration Verification Stock Standard Solutions are the source solutions to prepare the Initial Calibration Verification (ICV), and Continuing Calibration Verification (CCV) standards. Fresh mixed standards must be prepared as needed with the realization that concentrations can change on aging. Calibration standards must be initially verified using a quality control standard and monitored for stability.

Table 4. Calibration Verification Stock Standard Solutions (ICV/CCV)

Secondary Stock Solution	Elements	Conc. Stock (mg/L)	Vol. Stock (mL)	Final Volume (mL)	Final Conc. (mg/L)
A	Al, As, B, Ba Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Tl, V, Zn	1000 each	2.5 mL each	250	10
	Sb, Mo, Sn, Ti	1000 each	1.25	250	5
	Na, Ca, Mg, K, Fe	10,000 each	2.5 mL each	250	100
B	Ag	1000 each	2.5 mL each	250	10

10.11.1 Initial Calibration Verification Standard (ICV) - To a 100-mL volumetric flask add about 80 mL of 2% Nitric Acid (HNO_3). Pipette 0.5 ml of secondary stock solutions A and B into this flask and dilute to the mark with 2 percent (v/v) HNO_3 .

10.11.2 Continuing Calibration Verification Standard (CCV) – To a 250-mL volumetric flask add about 200 mL of 2% Nitric Acid HNO_3). Pipette 2.5 ml of secondary stock solutions A and B into a 250-ml flask and dilute to the mark with 2 percent (v/v) HNO_3.

10.12 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The method blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

10.12.1 The calibration blank consists of the same concentration(s) of the same acid(s) used to prepare the calibration standard solutions of the analytes (usually 2 % HNO_3 in reagent water).

10.12.2 The method (or digestion) blank must be carried through the complete digestion procedure and contain the same volumes of reagents as the sample solutions.

10.12.3 The rinse blank consists of 2% HNO_3 in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

10.13 Nitric Acid: Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Many more molecular-ion interferences are observed on the analytes when hydrochloric and sulfuric acids are used.

10.13.1 1:1 HNO_3: Cautiously and slowly add 100 mL concentrated HNO_3 to 100 mL of reagent water.

10.13.2 2% v/v HNO_3: Cautiously and slowly add 20 mL concentrated HNO_3 to a 1000 mL volumetric flask and bring to volume with reagent water and mix.

10.14 Argon gas supply: high-purity grade (99.99%).

11.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

11.1 The recommended containers used to collect samples for the determination of metals are polyethylene or fluorocarbon (TFE or PFA): 500 mL for water samples and 4 to 8 oz jars for soil samples.

11.2 Samples may be transported to the laboratory and stored at room temperature. Solid samples may be stored under refrigeration at between 0.1 and 6.0°C until time of digestion. Digestion and analysis must be within 180 days from sampling date. Sample digestates are stored at room temperature.

11.3 Aqueous samples must be preserved with 1:1 HNO_3 to a pH < 2.

11.4 For proper handling and storage of the PM10/SPM filters, see SOP 3115 Extraction of High Volume Filters.

12.0 QUALITY CONTROL

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality. The results of these QCI samples are used to assess the acceptability of data.

12.1 **Blanks**

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method blank shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second blank shall be prepared after the twentieth sample. The method blank shall be processed through all preparatory steps used for the

samples, including cleanup procedures. The blank shall be analyzed using the same instrument and conditions as the samples.

The Calibration Blank is used to monitor the initial level of contamination in the reagent solutions prior to initial calibration.

The Rinse Blank is used to monitor the ICP-MS system for potential cross-contamination from one sample to another or for residual contamination in the system.

12.2 **Initial Calibration Verification (ICV)**
An Initial Calibration Verification (ICV) standard containing all of the target analytes reported in this method (refer to section 10.11.1) shall be analyzed immediately after the completion of the initial calibration. The ICV shall be purchased from or prepared from second source standards to verify analyte concentrations.

12.3 **Continuing Calibration Verification (CCV)**
A CCV standard containing all of the target analytes reported in this method (refer to section 10.11.2) shall be analyzed after every tenth sample. The CCV standard shall be at a different concentration than the ICV, and shall be used to confirm the system is in calibration.

12.4 **Laboratory Control Sample (LCS)**
The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second LCS shall be prepared after the twentieth sample. The LCS shall be processed through all preparatory steps used for the samples, including cleanup procedures. The LCS shall be analyzed using the same instrument and conditions as the samples. Refer to section 10.10 for LCS analytes and concentrations.

12.5 **Duplicates**
Duplicates of field samples or of the LCS must be prepared in compliance with the method requirements and client directives. Note: the analysis of the Matrix Spike Duplicate (MSD) is used as a substitute for the laboratory duplicate. In those cases when there is insufficient sample to perform either a duplicate analysis or MSD analysis or the sample cannot be divided (e.g., wipes), the duplicate analysis of the LCS (LCS/LCSD) is used to judge the precision of the analytical results.

12.6 **Matrix Spike and Matrix Spike Duplicate (MS/MSD)**
MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire batch. MS/MSDs must be analyzed at a minimum of 1 per 20 samples per matrix per preparation procedure, or as specified by the required test method. If an MS/MSD pair is not analyzed in the preparation batch, an LCS/LCSD pair is analyzed. Samples chosen for matrix spiking are rotated among different clients and/or different client projects. This is accomplished through communication between the Department Manager and the analyst. In addition, designated samples, as indicated by client request or

contract requirement, are matrix spiked. The MS/MSD pair shall be processed through all preparatory steps used for the samples. They shall be analyzed using the same instrument and conditions as the samples. Refer to section 10.10 for MS analytes and concentrations. Some clients may require different spiking levels and/or target analytes. These specific needs are documented on the request for analysis forms.

12.7 **Internal Standards**

The internal standards for this method are used to monitor physical and matrix interferences and they are added to all samples, standards, and blanks. Refer to section 10.5 for preparation and concentration instructions.

12.8 **Interference Check Solutions**

The interference check solution (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on ^{51}V and $^{40}Ar^{35}Cl^{+}$ on 75As+. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. The ICS pair, solutions A and AB, is analyzed after calibration, every twelve hours of analysis, and at the end of the analytical batch. Refer to Section 10.9 for the preparation of these solutions.

Note: For ambient air filters, the ICS pair is analyzed every eight hours of analysis, and at the end of the analytical batch.

12.9 **Serial Dilutions**

If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the reagent blank, an analysis of a fivefold (1+4) dilution must agree within 10% of the original determination. If not, an interference effect must be suspected. One dilution test may be included for each twenty samples (or less) in a batch.

12.10 **Post-Digestion Spike**

This test may be applied for new or unusual matrices. An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125 %R of the known value or within the laboratory derived acceptance criteria. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. Results must agree to within 10% of the original determination. The use of a standard-addition analysis procedure may also be used to compensate for this effect.

13.0 CALIBRATION AND STANDARDIZATION

Calibrate the instrument after tuning to generate an acceptable Tune Report (section 14.2.1), and setting the Pulse/Analog factors for each analyte.

Note: Generating a tune report and setting the Pulse/Analog factors needs to be done only once each day. After calibration the software goes immediately into sample analysis. Step by step instructions to calibrate are listed in the procedure in section 14.

Initial Calibration (ICAL)
In addition to achieving the reference method requirements for the minimum number of calibration standards and the acceptance criteria (statistics) for calibration curve fit, the following ICAL criteria also apply:

13.1 The ICAL must be a minimum of two standards, not including a blank. Flush the system with the rinse blank between each standard solution. Use the average of at least three integrations for both calibration and sample analyses. All masses that could affect data quality should be monitored to determine potential effects from matrix components on the analyte peaks.
13.2 The ICAL must be verified with a second source standard (ICV) prior to the analysis of samples.
13.3 Results of samples not within the linear range of the instrument must be qualified on the final report. Dilute the sample and reanalyze in order to achieve a result within the linear range of the instrument.
13.4 The lowest calibration standard may establish the reporting limit: see Attachment 1 for analyte reporting limits (RL). The RL must be greater than the detection limit.

Initial Calibration Verification (ICV)
In addition to the method requirements, the following ICV criteria also apply:

13.5 Must be a second source standard from the ICAL standards or from a different manufacturer lot number.
13.6 Must be traceable to NIST when available.

13.7 Initial Calibration Blank (ICB): (2% HNO_3) Analyzed immediately after the ICV. Acceptance limits are ± RL.

Continuing Calibration Verification (CCV)
In addition to the method requirements, the following CCV criteria also apply:

13.8 Must be analyzed every 10 samples and at the end of each analytical batch.
13.9 Continuing Calibration Blank (CCB): (2% HNO_3 Analyze immediately after the CCV. Acceptance limits are ± RL.
13.10 If the CCV results obtained are outside the acceptance criteria, corrective actions must be performed. If routine corrective actions fail to produce an acceptable second consecutive CCV, then sample analyses cannot continue until a new ICAL is established and verified with an ICV.
NOTE: For calibration verification purposes, the CCV/CCB is analyzed as a set.

However, sample data associated with an unacceptable CCV may be reported as qualified data under the following special conditions:

13.10.1 When the acceptance criteria for CCV are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification must be reanalyzed after a new ICAL has been established, evaluated and accepted.

13.10.2 When the acceptance criteria for the CCV are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification must be reanalyzed after a new ICAL is established and verified with an ICV.

13.10.3 When the acceptance criteria for the CCV are exceeded and it is not possible to reanalyze the sample due to limited sample quantity AND a new sample cannot be obtained by the laboratory, the data may be reported with the appropriate data qualifiers if the client has been contacted and agrees, in writing, to accept the qualified data.

13.11 Records: Initial and Continuing Calibration Records will contain, at a minimum, the following:

1. Calibration date
2. Test method
3. Instrument
4. Analysis date
5. Each analyte name
6. Analyst's initials or signature
7. Standard Concentration (appropriate units) and number of standards
8. Response (appropriate units)
9. Calibration curve or response factor
10. Statistics for ICAL curve fit in order to judge calibration curve acceptance
11. Acceptance Limits for ICV analysis in order to judge calibration curve acceptance
12. Acceptance Limits for CCV analysis in order to judge continuing calibration acceptance
13. Calibration Standards and Reagent Solutions ID's

Table 5 Calibration Requirements

QC	Frequency	Standards	Control Limits	Corrective Action
ICAL	Daily or as needed	Minimum of two standards, see Table 2 for concentrations	r = 0.995	Correct problem then repeat initial calibration
ICV	After each new ICAL	25µg/L, 50 µg/L, 500 µg/L	± 10% of true value	Correct problem then repeat ICV or initial calibration
ICB	After each new ICV	Reagent Blank (2% HNO_3)	< RL	Correct problem then repeat ICB
CCV	Every 10 samples, and end of the batch	50 µg/L, 100 µg/L, 1000 µg/L	± 10% of true value	Correct problem then repeat CCV and associated samples, or repeat initial calibration
CCB	After each CCV	Reagent Blank (2% HNO_3)	< RL	Correct problem then repeat CCB and associated samples

Support Equipment
Autopipettes - Check autopipette to ensure standardization is within control limits (see SOP 1040 General Laboratory Practices for Pipette Calibration).

Dispensing Pipettes Check pipette to ensure standardization is within control limits (see SOP 1040 General Laboratory Practices for Pipette Calibration

14.0 PROCEDURE

14.1 Sample Preparation

14.1.1 Acid Digestion: Samples requiring digestion must be prepared by one of the following methods prior to ICP-MS analysis.

Matrix	SOPs
Water	3005
Soil/sediment/Waste	3110
TSP/PM10 filters	3115

14.1.2 Direct Analysis
For Dissolved Metals Analysis:
Filtering the Sample Through a 0.45 Micron Filter.
Wash the filter paper or filter disk with 5 mL of 2% nitric acid followed by 2-3 mL of sample before filtering the aliquot to be tested. Record this procedure in the comments section of the ICP-MS Preparations logbook. Take a 10 ml aliquot of the filtered sample and acidify with 0.2 mL of concentrated HNO_3. Filter a MB and LCS to be analyzed with the batch.

Also prepare a MS/MSD (if sample amount permits, otherwise prepare an LCS/LCSD), by spiking 10 mL of the filtered sample with 0.2 mL of LCS/MS stock (section 10.10) and analyze with the samples. Report results as dissolved metals.

For Drinking Water Analysis - Take a 10 ml aliquot of the sample and acidify with 0.4 mL of 1:1 HNO_3. Report results as total metals.

14.2 ICP-MS Analysis

Turn on the argon flow (100 psi minimum) from the Dewar. Turn on the water chiller. Connect all pump tubing. Double click ICP-MS Top icon. A message will appear "Is ISIS power on?" Turn it on if not already on. Click <yes>, <instrument>, <instrument control>, <plasma>, and <plasma on> to ignite the plasma and allow at least 30 minutes for warm up.

14.2.1 TUNING:
Click <ALS> in the instrument control screen, and <Go To>, to move the sipper probe to the tune solution. Put the internal standard tubing into reagent water. Close the Instrument Control Window. Open the tune screen by clicking <Instrument> and <Tune>. The tune window will open. Click <start> to monitor the counts per 0.1 seconds, and give a visual display of the RSD for counts for ^{7}Lithium, ^{89}Yttrium and ^{205}Thallium. The tune screen also displays the settings for

the Plasma Orientation, Gas Flows, Peripump RPMs, Lens settings, Q-Pole settings and Detector Parameters.

Tune the Agilent 7500 for sensitivity to ensure that the instrument produces the best results for the masses being analyzed. Achieve good sensitivity by running a recommended tuning solution of 10 parts per billion (ppb) of Li, Y, Ce and Tl. [ICP-MS Working Tuning Solution, 10.6]. If necessary adjust the tune parameters for maximum sensitivity, reducing Oxide Ions, and Doubly Charged Ions.

Operating conditions: Attachment 4 lists the affects of adjusting the parameters in the tune window on the tune of the instrument. Once you have adjusted the parameters satisfactorily, generate a Tune Report.

NOTE: A Tune Report MUST be generated prior to analyzing samples every day, whether the parameters have been changed or not.

TUNE SPECIFICATIONS:
Sensitivity:
Li > 5,000 cts/0.1sec @10 ppb concentration
Y > 10,000 cts/0.1 sec @ 10 ppb concentration
Tl > 5,000 cts/0.1sec @ 10ppb concentration

Precision:
Li < 15% RSD (0.1 sec integration time)
Y < 10% RSD (0.1 sec integration time)
Tl < 10% RSD (0.1 sec integration time)

Oxides: < 1.2 %

Double Charged: Ce ++/Ce+ < 5.0%

Background:
Li < 30 cps
Y <15 cps
Tl < 15 cps

Mass Resolution: W-10% 0.65 – 0.8 AMU

Mass Axis: nominal mass +/- 0.1 AMU for ^{7}Li, ^{89}Y and ^{205}Tl

After generating an acceptable tune report insert the sample tube for internal standards into the Internal Standard working solution and place the sipper probe for the sample line into 2% v/v HNO_3 rinse solution. Allow the system to rinse out the lines for at least five minutes.

14.2.2 SETTING P/A FACTORS:
ChemStation automatically switches between pulse and analog mode. For linear calibration curves, these two modes should be adjusted by using P/A Factor tuning.

The counts of each element must range from 400,000 to 4,000,000 cps to get accurate P/A Factors. The P/A factor adjustment must be performed everyday to get accurate results in a sample with a wide concentration range where both the pulse and analog mode are used.

Determine the P/A Factors by

1. Moving the sipper probe in the lowest concentration P/A solution.
2. After the solution has rinsed through into the plasma, click <tune> <adjust P/A factors>. 3. Click on <Load masses from acq. method> in the window that pops up.4. Click <run>. After about 20 seconds a message appears saying, "accept current P/A factors?" Check the clipboard window behind the message. Either a number (the P/A factor) or a message that states "sensitivity is too low" appears next to each mass. Click <yes> to accept the P/A factors and close out the clipboard window. If a message appears that states "sensitivity is too high" you can accept the P/A factor but will need to P/A with a lower concentration solution to get the P/A factor for the mass that gave "sensitivity too high."

Repeat steps 1, 2, and 4 for successively higher analyte concentrations until you have P/A factors for all masses of interest. Check the box "Merge in the current data" if not already checked. This will add in successive P/A factors without deleting the P/A factors for the masses, which have P/A factors already.

DO NOT REPEAT STEP 3 (click on <Load masses from acq. method> for each successive solution). This clears out all your P/A factors and should only be clicked on before you first start to P/A.

14.2.3 Load the appropriate method. (IO35.M for ambient airs samples, or 6020G.M for all other analyses.

14.2.4 TUNE CHECK:

Prior to calibration and analysis the tune conditions must be verified by analyzing a Tune Check Solution at least five times with relative standard deviations of < 5% for the analytes contained in the tuning solution.

Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria, which must be met prior to any samples being analyzed. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be less than 0.9 amu full-width at 10 percent peak height.

14.2.4.1 After tuning and P/A adjustments go to ICPMS top and click on <sequence>, <edit>, in the dropdown box on the left select TUNE, CCV, CALIB, or ICS, to edit the comment section in each of these screens. Enter any new solution preparation numbers for standards, ICV, CCV, ICB, ICS A, etc. in the comment column. Click <OK> at the bottom to save changes.

14.2.4.2 Click on <sequence>, <edit> again and select "SMPL" from the dropdown box to open up the sample sequence table. Edit inserting information under the column headings, VIAL (vial position), METHOD, SAMPLE (sample ID), COMMENT (sample test code), and Dil/Lv (dilution factor) for all samples to be analyzed. Click <print> to print out the list to refer to when placing the sample tubes in the racks and checking the order for correctness before starting analysis. Then click <save> to save the sample table. Samples are analyzed in order as they appear in the sample table.

14.2.4.3 Uncap and place all standards, tune check, and check standards in their proper places in rack #1. Racks #2-4 are for sample tubes.

14.2.4.4 Start the calibration and running of samples by clicking <sequence>, <run>. "Full Method'" and "Overwrite Existing Data Files," should be checked. Enter the instrument ID and your initials in the box for operator name and click <run sequence>. The software will run a blank, tune check, then the calibration followed by samples. After all samples have run the software will analyze a final ICS A, ICS AB, blank, CCV, and CCB. The plasma will then shut off and the ISIS program will stop. If additional samples will be analyzed later it will be necessary to insert blanks into the sample table to keep the instrument running.

14.2.4.5 Verify the Tune Check when complete. RSDs for Li, Co, In, and Tl must be < 5% and the mass calibration for each mass must be within 0.1 AMU of the mass number and <0.9 AMU at 10% peak height. If not abort the run and restart to rerun the Tune Check.

14.2.5 Initial Calibration and Quality Control Checks

14.2.5.1 Check each standard to determine if all internal standard cps are within 20% of the calibration blank's cps. If not abort the run and restart.

14.2.5.2 Check the ICV and ICB to determine if masses of interest are within tolerance. If they are continue with sample analysis. Rinse time before and after the samples are programmed into the method as well as analysis of CCV and CCB after every ten samples.

14.2.5.3 If an internal standard fails for any mass in a sample the system will run a blank before proceeding with analysis.

14.2.5.4 Perform all qualitative and quantitative measurements as described in Sections 14.4 and 14.5. After analysis, store the digests at room temperature.

14.2.6 The analytical sequence for Initial Calibration is:
(Tune Check)
Blank
Calibration Blank
Calibration standard (S1)
Calibration standard (S2)
Calibration standard (S3)
Calibration standard (S4)
Blank
Initial Calibration Verification Standard (ICV)

Initial Calibration Blank (ICB)
Interference Check Solution A (ICS A)
Interference Check Solution AB (ICS AB)
Blank

A typical analytical sequence for Sample Analysis is:
Method Blank (MB)
LCS
(LCS Duplicate optional)
Sample #1
Sample #1 Matrix Spike (MS)
Sample #1 Matrix Spike Duplicate (MSD)
Samples #2, #3, etc. up to 10 analytical samples
CCV
CCB
10 analytical samples (which may include, MB, LCS, MS, MSD, samples)
CCV
CCB
....
ICS A
ICS AB
blank
CCV
CCB

14.3 Data Interpretation - Qualitative Analysis

To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantification and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Unsuspected interferences may be detected by adding pure major matrix components to a sample to observe any impact on the analyte signals. When an interference source is present, the sample elements impacted must be flagged to indicate (a) the percentage interference correction applied to the data or (b) an uncorrected interference by virtue of the elemental equation used for quantitation. The isotope proportions for an element or molecular-ion cluster provide information useful for quality assurance.

NOTE: Only isobaric elemental, molecular, and doubly charged interference corrections that use the observed isotopic-response ratios or parent-to-oxide ratios are acceptable corrections for use in this method.

14.4 Data Interpretation - Quantitative Analysis

Calculations including appropriate interference corrections, internal-standard normalization, and the summation of signals at 206, 207, and 208 m/z for lead (to compensate for any differences in the abundances of these isotopes between samples and standards), are performed automatically by the instrument during analysis.

14.5 Records

Record the following information in the appropriate logbook or data file. Include any deviations from this procedure. Analyst initials, date of analysis, sample number or ID, initial sample volume or weight processed, calibration standard, sample, or solution identifier, QC sample or solution identifier, internal standard solution identifier, any dilution information, readings from support equipment, data file name, instrument method name, visual observations, and any other information as deemed necessary.

14.6 Troubleshooting

The sample line tubing and internal standard tubing should be checked for even flow when the instrument is first started by observing and air bubble passing through the line. Adjust the tension on the tubing so that it is just tight enough to allow the air to evenly move through the tubing without any pulsing.

Check for leaks and air bubbles at tubing connections, tee connectors and fittings. The majority of problems you encounter occur somewhere along the sample introduction path.

Ensure that the argon Dewar has enough argon to maintain a pressure of 100 psi throughout the run.

14.7 Routine Maintenance

Record all maintenance in the instrument logbook. For non-routine maintenance record the problem, what was done to correct it and whether the correction solved the problem.

14.7.1 Daily, or as needed according to sample throughput, examine the tubing
14.7.2 Clean the sample and skimmer cones on an as needed basis (loss of sensitivity).
14.7.3 Inspect the pump tubing for wear and replace as necessary. Check the Argon gas supply; replace as necessary.

14.8 Calibration of the Polypropylene Volumetric Flasks

(Yearly, independently check flask volume with reagent water using a balance to monitor weight of water volume: tolerance is stated volume ± 0.1%). Perform this check every year.

14.8.1 Initially check the volume of each flask. Using an analytical balance, record the weight of the empty and dry flask.
14.8.2 Add room temperature de-ionized reagent water.
14.8.3 Fill to mark and re-weigh.
14.8.4 Subtract initial weight from final weight.
14.8.5 Divide the result by the volume of the flask and multiply by 100.
14.8.6 The percent recovery should be ±0.1%.
14.8.7 Record this information and label the flask for reference.

15.0 DATA REDUCTIONS, CALCULATIONS, AND LOADING

15.1 The data system prepares a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

NOTE: The LIMS program will convert µg/L to the appropriate reporting units.

15.2 Report only those values that fall between the lowest calibration standard and the linear range of the instrument. Samples exceeding the linear range must be diluted and reanalyzed.

15.3 For sample results greater than the linear range, dilute the sample in a 15 mL centrifuge tube using 2% HNO_3. Pipette in the appropriate volume of sample into the tube and dilute to volume with 2% HNO_3. Dilution Factor = (10 mL/ digestate volume analyzed in mL). Use the dilution factor and calculate the concentration in the digestate as follows:

Concentration in mg/L = readout * 0.001 (10 mL/ digestate volume analyzed in mL)

15.4 Air Samples: calculate the concentration as follows:

Conc. in $\mu g/m^3$ = readout in µg/L * (10 mL/ initial sample air volume in m^3) * 1 L/1000 mL

NOTE: $1m^3$ = 1000 Liters

15.5 TSP/ PM10 Ambient Air Samples

15.5.1 Each filter should have an associated volume of air sampled, average temperature and pressure. This value is corrected to the EPA standard volume using the equation listed below.

$$V_{std} = V_{avg}(P_{avg}/760mm\ Hg)(298K/T_{avg})$$

Where:
V_{avg} is the calculated average flow rate during the elapsed sampling time (field data)
P_{avg} is the average barometric pressure in mm Hg during the sample run (field data)
T_{avg} is the average temperature in K during the sample run (field data)

15.5.2 Metals concentration in the air samples can then be calculated as follows:

$$C = [(\mu g\ metal/L) \times (Digestion\ volume,\ L/strip)(9) - F_m]/V_{std}$$

where:
C = concentration, µg metal/m3.
µg metal/L =determined metal concentration determined
final extract volume (L)/strip = total sample extraction volume from extraction procedure
9 = Useable filter area, [20 cm x 23 cm (8" x 9")]
Exposed area of one strip, [2.5 cm x 20 cm (1" x 8")].
F_m = average concentration of blank filters, µg.

V_{std} = standard air volume pulled through filter, std. m^3 (@25°C and 760 mm Hg).

15.5.3 When corrected average volume, average pressure and average temperature are NOT provided for a given filter, calculate µg metal/filter using the following equation

µg metal/ filter =[(µg metal/L) x (Digestion volume, L/strip)(9) - F_m]

15.5.4 Reported values should be blank subtracted for filters that STAT Analysis supplies to the client.

15.6 Wipe Samples: calculate the concentration as follows:

Conc. in µg/ft^2 = readout in µg/L * (50 mL/ initial wipe area in ft^2) * 1 L/1000 mL

NOTE: 1 ft^2 = 144 in^2 = 12 in * 12 in

15.7 Aqueous Samples: The concentration readout for aqueous sample digestates is µg/L. It does not need further data reduction unless the initial sample volume was less than 50 mL. If less than 50 mL sample was digested, calculate the concentration as follows:

Concentration in mg /L = readout *0.001 *(50mL/ initial sample volume digested in mL)

15.8 Soil Samples: The concentration readout for soil samples must be multiplied by the following factor: Factor = (50 / sample weight in g). Calculate the concentration in soil samples as follows:

Conc. in mg/Kg = readout * 0.001 (50 mL/ sample weight in g) * 1 L/1000 mL * 1000 g /1 Kg

15.9 Soil samples reported on a dry weight basis: The concentration is divided by the decimal equivalent of the percent residue of the soil after oven drying at 105 °C.

15.10 Report results in the appropriate units.

15.11 The procedure for uploading data into the LIMS system is detailed in SOP 1400 LIMS.

16.0 METHOD PERFORMANCE

Demonstration of Capability (DOC)
All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

A QC reference concentrate is required containing each analyte at a concentration of either 10 or 100 mg/L. The QC reference sample used is a ten-fold dilution of the CCV solution. The QC

reference sample is made using stock standards prepared independently from those used for calibration.

For each analyte calculate the mean recovery (X) and standard deviation (s) and the average % Recovery (%R). Compare X and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. X must be within 9/90 and 11/110 µg/L, respectively, and s must be less than 1.0/10.0 µg/L, respectively, and %R must be within 100 ± 10%.

These limits are taken from established in-house criteria. If X and s and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual X or %R falls outside the range for accuracy or any individual s exceeds the precision limit, then the system performance is unacceptable for that analyte and corrective action must be taken.

Comparison to Reference Method Data:
EPA Method 6020 Table 4, aqueous samples, provides guidance for the establishment of control limits for the LCS samples.

EPA Method 6020 Table 5, solid samples, provides guidance for the establishment of control limits for the LCS samples.

EPA Method IO-3.5, Table 7, solid samples, provides guidance for the establishment of control limits for the LCS samples.

In-House Control Limits: Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data is compared to the specifications of the reference method. If the in-house limits are within the specifications of the reference method, the control limits are updated in LIMS. If the in-house limits are not within specifications, an investigation is performed to determine the cause(s) of the problem and a corrective action is completed. The analysis may continue until enough data points are collected to regenerate new control limits. Any QC data generated outside of reference method limits during that time frame is flagged.

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

Control limits for the method parameters are generated by the QC staff and distributed to the analysts via updates to the LIMS control charts. The control limits are calculated based on in-house performance data. At a minimum, these limits are reviewed annually.

17.0 POLLUTION PREVENTION

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the lab waste disposal program, SOP 1130 Waste Disposal. With the consent of the client, the samples may be returned to their origin for treatment.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory staff is required to protect the laboratory and our clients' business information when disposing of recycled paper or waste from the facility.

18.0 DATA ASSESSMENT AND CRITERIA FOR QUALITY CONTROL MEASURES

Data assessment includes review of: proper sample condition, preservation, and storage; analysis within holding time limitations; deviations from the SOP, evaluation of performance based on in-house control limits, reference method limits or project specific limits.

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation. The data review is conducted according to SOP 1250 Data Review.

18.1 Blanks

If the Method Blank, ICB or CCB exceeds the RL the source of contamination must be investigated and corrective actions taken. The analyte concentrations in the method blank of a digestion batch must be less than the RLs listed in Attachment 1. If these criteria are exceeded, re-analyze the method blank. If after re-analysis, the blank criteria are still exceeded, then the entire digestion batch must be re-digested or qualified. Always refer to a client specific QAPP for additional guidance.

Affected samples must be reprocessed and reanalyzed or Data must be appropriately qualified if:

1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the SOP or by regulation, AND is greater than 1/10 of the amount measured in any sample.
2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

18.2 Laboratory Control Samples (LCS)

The results of the individual batch LCS are calculated in percent recovery (%R). Results are compared to established acceptance criteria: 100 ± 20% for all samples. If the LCS is outside the acceptance criteria the analytical system is "out-of-control." Any affected samples associated with an out of control LCS must be reprocessed and reanalyzed or the results reported with appropriate data qualifiers. If after re-analysis the control criteria have not been met, the entire digestion batch must be redigested for the out of control analytes of interest. Always refer to a client specific QAPP for additional guidance.

18.3 Duplicates

The results from laboratory duplicates are designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). See the STAT QAM, Section 5.4 for the calculation for RPD. Results are compared to established

acceptance criteria as listed in the MS/MSD. For duplicates results outside established criteria corrective action must be documented or the data reported with appropriate data qualifying codes.

For this test method, the analysis of the MS/MSD pair is used for determination of method precision. Duplicate LCS samples will be analyzed for all matrices where it would be impractical to perform a MS/MSD, such as wipe samples. Precision limits of 20% will be used for RPD acceptance criteria for MS/MSD and LCS/LCSD.

18.4 Matrix Spikes

The results from MS/MSD are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). See the STAT QAM, Section 5.4 for the calculation for RPD. Results are compared to established acceptance criteria: 100 ± 25%R and 20% RPD for all samples. For matrix spike results outside established criteria corrective action must be documented or the data reported with appropriate data qualifying codes. The RPD control limits are for analyte values greater than 100 times the instrumental detection limit. If this limit is exceeded, the reason for the out-of-control situation must be found and corrected.

NOTE: The %R limits will not apply to samples with analyte concentrations that are greater than four times the spike level.

Post Digestion Spike (PDS): This test may be applied for new or unusual matrices. An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125 %R of the known value or within the laboratory derived acceptance criteria. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. Results must agree to within 10% of the original determination. The use of a standard-addition analysis procedure may also be used to compensate for this effect.

18.5 Internal Standards

The results of the individual Internal Standard compounds on all samples, blanks, and spikes are calculated in percent recovery (%R) and are compared to established acceptance criteria. For instrument method 6020G.M, if the Internal Standard recovery in the test sample is outside the acceptance criteria, 30 to 120% of the response of the applicable internal standard in the ICAL Blank, corrective action must be taken. For ambient air samples, instrument method IO35.M, this limit is 60-125 %R. A dilution test (1+4) may be performed to minimize the matrix effect. Report the results from the diluted sample if internal standard recovery is acceptable.

NOTE: If the Internal Standard recovery in the calibration standards, ICV, ICB, CCV, or CCB is outside the acceptance criteria, 80 to 120% of the response of the applicable internal standard in the ICAL Blank, corrective action must be taken and affected samples must be reanalyzed.

18.6 Dilution Test

If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the reagent blank, an analysis of a fivefold (1+4) dilution must agree within 10% of the original determination. If not, an interference effect must be suspected. One dilution test may be included for each twenty samples (or less) in a batch.

18.7 ICS

Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run and once every 12 hours for instrument method 6020G.M. For ambient air sample, instrument method IO35.M, the ICS pair is analyzed at the beginning of the analytical run and once every eight hours. The ICS pair is analyzed at the end of the run as well for both methods. Do this by analyzing the interference check solutions A and AB. The analyst should be aware that precipitation from solution AB may occur with some elements, specifically silver. The recovery of each element in the ICS solution should be 100 ± 20%.

18.8 Instrument Detection Limits

IDLs in µg/L can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least every three months and kept on file.

19.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the ICV, ICB, CCV, CCB, MB, LCS/LCSD, MS/MSD, PDS, lab duplicate, or internal standard recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrective action taken. The analytical result for that parameter in the samples is suspect and is only reported for regulatory compliance purposes with the appropriate corrective action. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. Final data results must be qualified in the client report for reported results not meeting the laboratory-defined criteria.

1) Review standards preparation logbooks. Check all calculations and ensure dilution factors are properly recorded.
2) Re-prepare the suspected standard or QC sample to identify possible preparation errors of the standard or QC sample.
3) Re-Analyze the samples when the CCV or LCS is not within acceptable limits.
4) Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

20.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director will review data and discuss options with the client. Reanalysis or reporting the data with qualification are alternatives. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.
Holding time exceedence, improper preservation and improper sample condition or storage are noted on the corrective action form and included on the final report.
Review the CCV standard response, LCS result, ICS results, and internal standard recovery for acceptable performance for each batch of samples. Record any trends or unusual performance on a corrective action form. Final data results must be qualified in the client report for results not meeting the laboratory-defined criteria.

20.1 The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

20.2 Client Requested Modifications:
- 20.2.1 Clients must request modifications from the laboratory SOP in writing to the lab.
- 20.2.2 The lab director, technical manager and quality assurance manager will evaluate the requested client deviations, determine the feasibly of the deviation and the potential effects on the data.
- 20.2.3 If it is determined that the lab will perform the requested deviations, lab director, technical manager and quality assurance manager will decide if a method validation study is required.
- 20.2.4 The designated project manager will retain all documentation concerning the requested deviation, including all correspondence with the client, in the client folder.
- 20.2.5 The final analytical report must include the statement "This report has analyses performed using client requested modifications."

21.0 WASTE MANAGEMENT

The STAT Analysis Corporation Waste Disposal SOP 1130 identifies proper waste management practices for the chemicals and biolo gical materials used in this procedure. Samples are stored and discarded accordance with SOP 1130 Waste Disposal.

22.0 REFERENCES

22.1 Method 6020, Revision 0, December 1996; U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" Update III, December 1996

22.2 Compendium Method IO-3.5 Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air: Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/ Mass Spectrometry (ICP/MS), U.S. EPA, June 1999.

22.3 STAT Analysis Corporation Quality Assurance Manual
22.4 STAT SAP 003 Chemical Hygiene Plan
22.5 STAT SOP 230 Corrective Actions
22.6 STAT SOP 1000 Control and Use of Laboratory Notebooks
22.7 STAT SOP 1010 Standard and Reagent Preparation
22.8 STAT SOP 1040 General Laboratory Procedures
22.9 STAT SOP 1130 Waste Disposal
22.10 STAT SOP 1210 Method Detection Limits (MDLs)
22.11 STAT SOP 1230 Training
22.12 STAT SOP 1250 Data Review
22.13 STAT SOP 1400 LIMS
22.14 STAT SOP 3005 SW846 3005A Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA, ICP, or ICP-MS
22.15 STAT SOP 3110 SW846 3050B Acid Digestion of Sediments, Sludges, and Soils for Metals Analysis by FLAA, ICP, or ICP-MS
22.16 STAT SOP 3115 Acid Digestion of High Volume Filters
22.17 Manufacturers' Equipment Instruction Manuals

23.0 FORMS, FIGURES, TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS OR VALIDATION DATA

Attachment 1: MDLs and Reporting Limits
Attachment 2: Concentrations of ICS Solutions
Attachment 3: Concentrations of Spike Solutions
Attachment 4: Tune adjustment Parameters

ATTACHMENT 1: MDLs and REPORTING LIMITS FOR ELEMENTS

				Aqueous		Soil		Air	
CAS #	Element	Mass (Isotope) *, **	IDL (µg/L)	MDL (mg/L)	RL (mg/L)	MDL (mg/Kg)	RL (mg/Kg)	MDL (µg/ filter)	RL (µg/ filter)
7429-90-5	Aluminum	27	0.235	0.00309	0.02	0.3271	2	NA	NA
7440-36-0	Antimony	**121***, 123	0.606	0.00249	0.003	0.1099	0.2	0.75	5
7440-38-2	Arsenic	75	0.063	0.00097	0.002	0.0429	0.1	0.69	5
7440-39-3	Barium	135, **137***	0.007	0.00140	0.002	0.0260	0.1	27	200
7440-41-7	Beryllium	9	0.004	0.00027	0.001	0.0074	0.05	0.25	5
7440-43-9	Cadmium	**111***, 114	0.015	0.00021	0.001	0.0078	0.05	0.20	5
7440-47-3	Chromium	**52***, 53	0.027	0.00054	0.002	0.0260	0.1	1.4	10
7440-48-4	Cobalt	59	0.019	0.00011	0.002	0.0051	0.1	0.18	5
7440-50-8	Copper	**63***, 65	0.080	0.00158	0.005	0.0404	0.25	0.52	5
7439-92-1	Lead	**206,207, 208****	0.019	0.00059	0.001	0.0065	0.05	0.40	5
7439-96-5	Manganese	55	0.020	0.00029	0.002	0.0114	0.1	0.82	10
7440-02-0	Nickel	**60***, 62	0.186	0.00107	0.002	0.0434	0.1	0.54	10
7782-49-2	Selenium	82	0.210	0.00172	0.002	0.0646	0.1	0.44	5
7440-22-4	Silver	**107***, 109	0.050	0.00079	0.002	0.0278	0.1	0.071	5
7440-28-0	Thallium	205, **203***	0.096	0.00026	0.002	0.0080	0.1	0.17	5
7440-32-6	Titanium	46, 47*		0.00057	0.005	.0261	0.25	1.1	10
7440-66-6	Zinc	**66***, 67, 68	0.080	0.00160	0.01	0.1157	0.5	2.2	25
7440-23-5	Sodium	23	0.671	0.07706	0.15	1.0965	6	NA	NA
7439-95-4	Magnesium	**24***, 25	0.193	0.00832	0.05	0.4429	3	NA	NA
7440-09-7	Potassium	39	0.585	0.03922	0.05	0.7043	3	NA	NA
7440-70-2	Calcium	44	2.698	0.07448	0.10	6.3708	6	NA	NA
7440-62-2	Vanadium	51	0.009	0.00087	0.002	0.0959	0.1	0.52	5
7439-89-6	Iron	**57***, (54)	0.611	0.01154	0.05	0.6888	3	4.6	50
7439-98-7	Molybdenum	92,94,96,97, **98***	0.020	0.00033	0.005	0.0405	0.5	0.37	5
7440-31-5	Tin	**120***, 118	0.026	0.00054	0.01	0.0772	0.5	0.24	5
7440-42-8	Boron	**11***, 10	.332	0.00551	0.02	0.2322	1	NA	NA

NOTES:

* Primary mass used for quantitation. Other masses are optionally used if proper instrument QC is available.

** The summation of all three lead masses is used for quantitation.

NA = not applicable

ATTACHMENT 2: CONCENTRATIONS of ICS SOLUTIONS

Solution A: Concentration in mg/L

Element	Stock	Working
Al	500	100
C	1000	200
Ca	500	100
Cl	3600	720
Fe	500	100
K	500	100
Mg	500	100
Mo	10	2
Na	500	100
P	500	100
S	500	100
Ti	10	2

Solution AB: Concentration in mg/L

Element	Stock	Working		Element	Stock	Working
Al	500	100		Ag	0.100	0.020
C	1000	200		As	0.100	0.020
Ca	500	100		Cd	0.050	0.010
Cl	3600	720		Co	0.200	0.040
Fe	500	100		Cr	0.100	0.020
K	500	100		Cu	0.100	0.020
Mg	500	100		Mn	0.100	0.020
Mo	10	2		Ni	0.200	0.040
Na	500	100		Se	0.100	0.020
P	500	100		V	0.200	0.040
S	500	100		Zn	0.100	0.020
Ti	10	2				

ATTACHMENT 3: CONCENTRATIONS OF LCS/MS SPIKING SOLUTION

Element	Concentration (mg/L)
Aluminum	25
Antimony	12.5
Arsenic	25
Barium	25
Beryllium	25
Boron	25
Cadmium	25
Chromium	25
Cobalt	25
Copper	25
Lead	25
Manganese	25
Molybdenum	12.5
Nickel	25
Selenium	25
Silver	10
Thallium	25
Titanium	12.5
Tin	12.5
Vanadium	25
Zinc	25
Calcium	100
Iron	100
Magnesium	100
Potassium	100
Sodium	100

ATTACHMENT 4: Tune Adjustment Parameters

Plasma conditions:
Higher RF Power generally increases the sensitivity.

Sample Depth
Shortening sample depth increases sensitivity and raises oxide levels.

To reduce Oxide Ions <1.2%

- Increase the sampling depth
- Decrease the carrier gas flow
- Increase the RF power
- Decrease the sample flow rate

To Reduce the Doubly Charged Ions < 5%

- Increase the sampling depth
- Decrease the carrier gas flow
- Increase the RF power
- Decrease the sample flow rate

Resolution and Mass Axis
Peak width at 10 % should be 0.65 – 0.8 AMU
Mass Axis should be within +/- 0.1 AMU of the selected mass

AMU Gain - Adjusts the peak width. The higher the value the narrower the peak width for heaver masses

AMU Offset - Adjusts the peak width. The higher the value the narrower the peak width for ALL masses

Axis Gain - Adjusts mass calibration. Higher value shifts the peak of heavier masses towards higher mass

Axis Offset - Adjusts mass calibration. Higher value shifts the peak of ALL masses towards higher mass

QP Bias - Controls the speed of ions as they pass through the Q-Pole.

SOP ADDENDUM

SOP No. & TITLE: No. 4510. Metals analysis by ICP-MS.

Revision Effective Date: 05. May 16, 2005.

Issued by Pinaki Banerjee, QA Director

Approved by: Dennis Jachim, Technical Manager

Date of Issue: August 6, 2007

Section (additions are *italicized*, ~~deletions are strikethroughs~~)

14.2.1

TUNE SPECIFICATIONS:
Sensitivity:
Li > 5,000 cts/0.1sec @10 ppb concentration
Y > 10,000 cts/0.1 sec @ 10 ppb concentration
Tl > 5,000 cts/0.1sec @ 10ppb concentration

Precision:
Li < 15% RSD (0.1 sec integration time)
Y < 10% RSD (0.1 sec integration time)
Tl < 10% RSD (0.1 sec integration time)

Oxides: < 1.2 % *(Babington Nebulizer)*
< 2 % *(Concentric Nebulizer)*

Double Charged: Ce ++/Ce+ < 5.0%

Attachment 4

Plasma conditions:
Higher RF Power generally increases the sensitivity.

Sample Depth
Shortening sample depth increases sensitivity and raises oxide levels.

To reduce Oxide Ions <1.2% *(< 2% for concentric nebulizer)*

- Increase the sampling depth
- Decrease the carrier gas flow
- Increase the RF power
- Decrease the sample flow rate

SOP ADDENDUM

SOP No. & TITLE: 4510, Metals Analysis by ICP

Revision 05; Effective Date: May 16, 2005.

Issued by : Pinaki Banerjee, QA Director

Approved by: Dennis Jachim, Technical Manager

Date of Issue: March 14, 2008

Section (additions are *italicized*, ~~deletions are strikethroughs~~)

5.3 Method Modifications from Reference – This SOP is based on EPA Method 6020 and incorporates IO-3.5 for TSP/PM10 high-volume filters except that: 1) polypropylene volumetric flasks are used since analysis for boron is occasionally required, 2) the instrument resolution is set for 1 amu peak width at 10% peak height according to 6020 but is less stringent than IO-3.5, 3) samples which fail high or low for one or more internal standards are diluted as needed to bring within range for the internal standard, 4) A matrix spike and matrix spike duplicate are digested and analyzed, since a duplicate sample is not typically analyzed, 5) ~~Dilution test may be performed on aqueous matrices. Non aqueous matrices are already diluted before analysis~~ *The dilution test will be applied to preparation batches /matrices in which analyte concentration is at least 10 times greater than the reporting limit.* 6) reagent water must be at least Type II (ASTM) for all standards, solutions, and sample preparation according to 6020 but is less stringent than IO-3.5, 7) internal standards and tuning solutions cover the entire mass range of analysis, but the elements used vary slightly from IO-3.5, 8) the MDLs are determined annually according to the requirements described in SOP 1210 Method Detection Limits ~~and not every six months according to IO-3.5,~~ 9) the ICB/CCB acceptance limit is the ~~lower~~ reporting limit and not the MDL per IO-3.5, since we do not report down to the MDL. 10) Germanium, instead of yttrium, is used as an internal standard, since yttrium is found in environmental samples.

12.9 **Serial Dilutions**

If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 10 ~~100~~ times greater than the ~~concentration in the~~ *reporting limit* ~~reagent blank~~, an analysis of a fivefold (1+4) dilution must agree within 10% of the original determination. If not, an interference effect must be suspected. ~~One dilution test may be included for each twenty samples (or less) in a batch.~~

18.6 **Dilution Test**

If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least ~~100~~ 10 times greater than the ~~concentration in the reagent blank~~ *reporting limit*, an analysis of a fivefold (1+4) dilution must agree within 10% of the original determination. If not, an interference effect must be suspected. One dilution test ~~may~~ *must* be included for each twenty samples (or less) in a batch.

STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 4715

AUTOMATED PHENOLS-4AAP ANALYSIS By EPA 9066

Revision 01
Effective Date: June 6, 2005

Authors: Ian Graske and Bruce Anderson

Printed Name	Signature/Date
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This Standard Operating Procedure has been prepared for the sole use of STAT Analysis Corporation.

Copy Number: _____

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Table of Contents

1.0 Identification of Test Method

SOP Title: Automated Phenols-4AAP Analysis by EPA 9066 is also known as Phenols-4AAP in the laboratory records.

2.0 Applicable Matrix or Matrices

This method is used to determine the concentration of phenols in aqueous samples, wastes, and leachate. This method is used to quantify the concentration of phenols from the distillation procedure detailed in STAT SOP 3620 Phenolics Distillation by EPA Method 9066.

3.0 Detection Limits

The lab follows the procedure found in 40CFR Part 136B to determine the MDL for each matrix type on an annual basis. See the STAT Analysis SOP 1210 Method Detection Limits for the MDL procedure, frequency and acceptance criteria. The MDL's measured by the lab and all supporting documentation are in the laboratory QA files for review.

The laboratory determined MDL must always be less than the reporting limit (RL). The RL will usually range from three to ten times the laboratory measured MDL but this relationship may vary dependent on dilution of sample aliquots, matrix interferences, moisture adjustments (in solid samples), or method-specified requirements.

The present MDL for aqueous samples is 0.002 mg/L. The present MDL for soils is 0.1 mg/kg. The applicable reporting limit range for aqueous samples is 0.005 mg/L. The applicable reporting limit for soil samples is 0.25 mg/Kg (as received basis). Sample distillates with concentrations greater than the highest calibration standard are diluted and then reanalyzed. Samples with high concentrations of phenols may also be redistilled using a smaller sample size and then analyzed.

4.0 Scope and Application

The method is designed for the analysis of aqueous distillates for phenols. Samples and QC samples are distilled prior to analysis.

The distillation procedure described in SOP 3620 Phenolics Distillation by EPA 9065 is designed for the determination of phenols in aqueous solutions, solid waste materials, or effluents. This distillation method is not applicable to oil or multiphasic samples or samples not amenable to the distillation procedure.

NOTE: It is not possible to use this method to differentiate between different kinds of Phenols. Phenol, ortho- and meta- substituted phenols, and para- substituted phenols where the para- group is a carboxyl, a halogen, a methoxyl, or a sulfuric acid group are all determined by reaction with

4-aminoantipyrine. Not determined are para-cresol, and other para- substituted phenols where the para- group is an alkyl, an aryl, a nitro, a benzoyl, a nitroso, or an aldehyde group.

Color response of phenolic materials with 4-AAP is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason, phenol (C_6H_5OH) itself has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.

This method is restricted to use by or under the supervision of analysts experienced in the use of the LaCHAT AutoAnalyzer.

Note: Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 Summary of Method

The method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide ($K_3Fe(CN)_6$) and 4-amino-antipyrine (4-AAP) to form a red complex that is measured from 500 to 520 nm. This solution is analyzed on the LaCHAT AutoAnalyzer.

Method Modifications from Reference

1. This SOP reflects the reduced volume version of the method. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.
2. The duplicate is analyzed once every 20 samples in accordance with NELAC.
3. Sulfuric acid is not added to standards. Interferences from sulfur compounds and biological degradation are not issues. Furthermore, the QC indicates that the system is in control. The pH of the samples is 7, after the distillation.

6.0 Definitions

The STAT Analysis Corporation Quality Assurance Manual (QAM) contains the definitions of standard terms used in this SOP.

7.0 Interferences

7.1 Interferences are eliminated or reduced by using the distillation procedure described in SOP 3620 Phenolics Distillation by EPA 9065.

7.2 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of <4.0 with H_2SO_4 and aerating briefly by stirring.

7.3 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of

an excess of ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

7.4 Background contamination from plastic tubing and sample containers is minimized by using non-reactive plastic ware.

7.5 DURAPRENE PUMP TUBES MUST BE USED WITH THIS METHOD.

7.6 Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

8.0 Safety

8.1 All samples must be assumed as hazardous and appropriate precautions taken during handling.

8.2 Safety glasses, gloves, lab coats and closed toe shoes are to be worn.

8.3 Other safety precautions must be conducted in accordance with the Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of material safety data sheets (MSDS) is available in each room for personnel involved in an analysis using chemicals.

8.4 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

8.5 The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consult the MSDS.

8.5.1 Phenol is toxic and hygroscopic. Use extreme caution when handling this material.

8.5.2 Sulfuric acid is a strong oxidizer. Use extreme caution when handling this material. Avoid eye and skin contact. Wash exposed areas immediately with copious amounts of water.

8.5.3 Potassium Ferricyanide is hazardous. Use extreme caution when handling this material.

9.0 Equipment and Supplies

9.1 LaCHAT AutoAnalyzer consisting of the following components:

9.1.1 Autosampler Cetac

9.1.2 Reagent Pump

9.1.3 System Unit LaCHAT 8000

9.1.4 Computer

9.1.5 Printer

9.2 Centrifuge Tubes, 50 mL graduated

9.3 Volumetric Flasks, Class A: 1000mL, 250mL, 100mL, 50mL, 25mL with stoppers

9.4 Autopipetter: 0.010 to 0.10 mL, 0.10 to 1.0 mL, 1.0 to 5.0 mL

9.5 Test Tubes, 15 mL and Tube Racks

9.6 Plastic and glass bottles for solution storage

9.7 Lead Acetate Paper

10.0 Reagents and Standards

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see SOP 230 Corrective Action).

10.1 Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagents Receipt and Preparation.

10.2 Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used.

10.3 Use reagent water for all solutions. Degassing with helium: If necessary to prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (LaCHAT Part No. 50100.) Bubble He through the solution for one minute.

10.4 4-Amino-antipyrine Color Reagent: In a 250 mL volumetric flask dissolve 0.16 g 4-aminoantipyrine (Aldrich A3, 930-0, Sigma A4382, or equivalent) in 250 mL DI water. Store in glass and prepare fresh daily.

10.5 Buffered Potassium Ferricyanide, pH 10.3: In a 1 L volumetric flask, dissolve 2.0 g potassium ferricyanide [$K_3Fe(CN)_6$], 3.1 g boric acid (H_3BO_3) and 3.75 g potassium chloride (KCl) in about 800 mL DI water. Add 47 mL 1 M sodium hydroxide and dilute to the mark with DI water. Shake to mix. Store in glass and prepare fresh weekly.

10.6 1 M Sodium Hydroxide Solution (stock): In a 500 mL volumetric flask, dissolve 20 g sodium hydroxide (NaOH) in 250 mL reagent water, cool and dilute to mark with reagent water.

10.7 Standards

10.7.1 At least one of the standards must be traceable to a NIST traceable source when available. The manufacturer should include a certificate of analysis for each standard. If one is not provided, contact the manufacturer. Retain all certificates in the designated binder (see SOP 1010 Analytical Standards and Reagents Receipt and Preparation.)

10.7.2 Standards must be prepared volumetrically using class-A volumetric glassware, calibrated micropipettes, or gas tight syringes. Do not use disposable pipettes to prepare standards.

10.7.3 Phenol Stock Calibration Standard: 1000 mg/L Commercially Purchased. Store per manufacturer's recommendations and shelf life. If shelf life not stated, then this solution may be used for twelve months if stored in the original container at 4°C and shows no sign of deterioration.

10.7.4 Intermediate Phenol Calibration Solution: (10 mg/L or 10 µg/mL): Dilute 1 mL of Phenols Stock Standard to 100 mL reagent water. Prepare this solution fresh monthly and store in a glass-stoppered bottle at 4°C.

10.7.5 Phenol Working Calibration Standards. Dilute the Intermediate Phenols Calibration Solution to 50 mL with reagent waterto make the following Calibration Standards: See Table 1. Prepare these solutions fresh daily and store in 50 mL graduated tubes.

10.7.6 Stock ICV/CCV Phenol Standard: 1000 mg/L Phenol commercially purchased from a second source. (Different Lot or Different Vendor) Store per manufacturer's recommendations and shelf life. If shelf life not stated, then this solution may be used for twelve months if stored in the original container at 4°C and shows no sign of deterioration.

10.7.7 Intermediate ICV/CCV Standard Solution: (10 mg/L or 10 µg/mL): Dilute 1 mL of Phenols Stock ICV/CCV to 100 mL reagent waterPrepare this solution fresh monthly and store in a glass-stoppered bottle at 4°C.

10.7.8 Working ICV Solution 50 µg/L (0.05 mg/L): Dilute 0.25 mL of Intermediate ICV/CCV Standard to 50 mL reagent water. Prepare this solution fresh at least weekly and store in a 50 mL graduated tube at 4°C.

10.7.9 Working CCV Solution 100 µg/L. Dilute 0.50 mL of Intermediate ICV/CCV Standard to 50 mL with reagent water. Prepare fresh at least weekly and store in a 50 mL graduated tube at 4 °C.

The daily calibration standards are prepared as follows (use autopipetters and flasks as listed in Section 9). Add the listed volume of the Intermediate Phenols Calibration Solution to each tube, dilute to mark with reagent water, cap and mix well.

Table 1 Phenols Calibration Standards

Calibration Standard Concentration, µg/L	Amount of solution to add to volumetric, mL	Concentration of solution added to volumetric, µg/mL	Final Volume, mL	µg Phenol per 50 mL
200	1.0	10	50	10
100	0.5	10	50	5.0
50	0.25	10	50	2.5
10	0.050	10	50	0.50
5.0	0.025	10	50	0.25
0	0	0	50	0

11.0 Sample Collection, Shipment, Preservation and Storage

See SOP 3620 Phenolics Distillation by EPA 9065 for details. Samples shall be placed on ice immediately after collection. The holding time is 28 days for a refrigerated sample (4°C) with proper chemical preservation (pH < 4). Distillation and analysis must occur within the 28-day period to be compliant.

Distilled samples may be stored at room temperature (or refrigerated) for up to one month.

12.0 Quality Control

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality.

The results of these QCI samples are used to assess the acceptability of data.

12.1 Blanks

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method blank shall be prepared once per preparation batch of 20 or less samples per matrix type (see Section 6 for definition of a prep batch). If more than 20 samples are prepared a second blank shall be prepared after the twentieth sample. The method blank shall be processed through all preparatory steps used for the samples, including cleanup procedures. The blank shall be analyzed using the same instrument and conditions as the samples.

12.2 Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS shall be prepared once per preparation batch of 20 or less samples per matrix type. If more than 20 samples are prepared a second LCS shall be prepared after the twentieth sample. The LCS shall be processed through all preparatory steps

used for the samples, including cleanup procedures. The LCS shall be analyzed using the same instrument and conditions as the samples. Refer to SOP 4620 for preparation and concentration instructions.

12.3 Duplicates

Duplicates of field samples must be prepared every 20 samples in compliance with the NELAC standard and/or with client directives. Note: the analysis of the Matrix Spike Duplicate (MSD) is used as a substitute for the laboratory duplicate

12.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire batch. MS/MSDs must be analyzed at a minimum of 1 per 20 samples per matrix per preparation procedure, or as specified by the required test method. For cases where the sample cannot be divided (e.g., wipes, air samples, not enough sample provided by customer) and thus a MS/MSD pair cannot be prepared for in the preparation batch, an LCS/LCSD pair is analyzed to measure precision and accuracy.

Samples chosen for matrix spiking are rotated among different clients and/or different client projects. This is accomplished through communication between the Department Manager and the analyst. In addition, designated samples, as indicated by client request or contract requirement, are matrix spiked.

The MS/MSD pair shall be processed through all preparatory steps used for the samples. They shall be analyzed using the same instrument and conditions as the samples. Refer to SOP 3620 Phenolics Distillation by EPA 9065 for preparation and concentration instructions. Some clients may require different spiking levels; these specific needs are documented on the request for analysis forms.

13.0 Calibration and Standardization

Initial Calibration (ICAL)

In addition to achieving the reference method requirements for the minimum number of calibration standards and the acceptance criteria (statistics) for calibration curve fit, the following ICAL criteria also apply:

13.1 The ICAL must be a minimum of 5 standards.

13.2 The ICAL must be verified with a second source standard (ICV) prior to the analysis of samples.

13.3 Results of samples not bracketed by the ICAL range must be qualified on the final report. If possible, dilute the sample or extract and reanalyze in order to achieve a result within the calibrated range of the instrument.

13.4 The lowest calibration standard establishes the reporting limit: RL = .005mg/L for waters and 0.25 mg/Kg for soils (as received basis). The RL must be greater than the detection limit.

Initial Calibration Verification (ICV)

In addition to the method requirements, the following ICV criteria also apply:

13.5 Must be a second source standard from the ICAL standards or from a different manufacturer lot number.

13.6 Must be traceable to NIST when available.

13.7 Must be analyzed when an ICAL is not performed on the day of analysis, prior to sample analysis.

Continuing Calibration Verification (CCV)

In addition to the method requirements, the following CCV criteria also apply:

13.8 May be analyzed at the beginning of the batch to check the CCV recovery.

13.9 Must be analyzed after every 10 samples and at the end of each analytical batch.

13.10 If the CCV results obtained are outside the acceptance criteria, corrective actions must be performed. If routine corrective actions fail to produce an acceptable *second consecutive (immediate)* CCV, then either the lab has to demonstrate performance after corrective action with two consecutive successful CCVs, or a new ICAL must be performed. If the instrument has not demonstrated acceptable performance, sample analyses cannot continue until a new ICAL is established and verified with an ICV. However, sample data associated with an unacceptable CCV may be reported as qualified data under the following special conditions:

13.10.1 When the acceptance criteria for CCV are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification must be reanalyzed after a new ICAL has been established, evaluated and accepted.

13.10.2 When the acceptance criteria for the CCV are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification must be reanalyzed after a new ICAL is established and verified with an ICV.

13.10.3 When the acceptance criteria for the CCV are exceeded and it is not possible to reanalyze the sample due to limited sample quantity AND the laboratory cannot

obtain a new sample, the data may be reported with the appropriate data qualifiers if the client has been contacted and agrees, in writing, to accept the qualified data.

13.11 Records: Initial and Continuing Calibration Records will contain, at a minimum, the following:

1. Calibration date
2. Test method
3. Instrument
4. Analysis date
5. Each analyte name
6. Analyst's initials or signature
7. Standard Concentration (appropriate units) and number of standards
8. Response (appropriate units)
9. Calibration curve or response factor
10. Evaluation of and Statistics for ICAL curve fit in order to judge calibration curve acceptance
11. Evaluation of and Acceptance Limits for ICV analysis in order to judge calibration curve acceptance
12. Evaluation of and Acceptance Limits for CCV analysis in order to judge continuing calibration acceptance
13. Calibration Standards and Reagent Solutions IDs

Calibration Acceptance Summary (see Table 2):

Table 2 Calibration Requirements

QC	Frequency	Standards	Control Limits	Corrective Action
ICAL	Daily or as needed, minimum monthly	Minimum 5 standards, see Table 1 for concentrations	$r^2 = 0.99$, or $r = 0.995$	Correct problem then repeat initial calibration
ICV	After each new ICAL And at the beginning of each new run.	50 µg/L	± 10% of true value	Correct problem then repeat initial calibration
ICB	After each new ICV	Reagent Blank	< RL	Correct problem then repeat ICB
CCV	Beginning (optional), every 10 samples, and end of the batch	100 µg/L	± 10% of true value	Correct problem then repeat CCV or repeat initial calibration
CCB	After each CCV	Reagent Blank	< RL	Correct problem then repeat CCB and associated samples

Support Equipment

Autopipettes - Check autopipette to ensure standardization is within control limits (see SOP 1040 for Pipette Calibration).

Balances - Be sure the balance is checked prior to use and performance criteria are met (see SOP 1040 for Calibration of Balances).

14.0 Procedure

14.1 Instrument Start Up

14.1.1 Turn on the power to all modules by turning on the power strip and allow the autosampler to perform its startup routine. Wait until the autosampler stops with the probe above the wash bath.

14.1.2 If necessary, install the manifold on the channel you want to run. (See Appendix E.)

14.1.3 Make all the injection fluidic connections.

14.1.4 Make all the cell fluidic connections.

14.1.5 Set all pump tubes on the pump. (Varies method to method.)

14.1.6 Run reagent water through all the lines to make sure there are no leaks.

14.1.7 If there are no leaks, put actual reagents in line.

14.1.8 ALWAYS start pumping Buffer Solution FIRST.

14.1.9 Pour the calibration Standards into Standard vials, and place into the autosampler Standards Rack.

14.1.10 Pour the Samples into test tubes and place into the Sample Rack.

14.1.11 If necessary, insert the interference filter into the upper slot in the detector.

14.2 Software Set up

14.2.1 Double click on the **Omnion FIA** icon. Then click **OK**. The autosampler probe should go into the wash bath and the dilutor activity may be heard, the injection valves may turn to the inject state if they were not already there.

14.2.2 Log In with your user name and password. [User name: demo with no password will also get you in]. The Main Menu should appear.

14.2.3 From the Main Menu click on the Instrument button labeled **FIA Instrument 1.** Each valve allocated to this instrument will be cycled in turn from load to inject and back to inject again. Omnion will automatically open the last Method and TRAY that was used. (The names will appear at the top in the title bar.)

14.3 Open the Phenols Method (To Create a Method see Appendix B)

14.3.1 Click on the **Method** button, or from the Main Menu click on **File**, then **Open Method**. This will open the Open Method Dialog window.

14.3.2 Double click on the method file you want to open: **Phenols.met** Or click once then click on **OK**.

14.3.3 From the Main Menu click on the Instrument button labeled **FIA Instrument 1.** Each valve allocated to this instrument will be cycled in turn from load to inject and back to inject again. Omnion will automatically open the last Method and TRAY that was used. (The names will appear at the top in the title bar.)

14.4 Opening and Editing the TRAY

14.4.1 Click on the **TRAY** button or from the Main Menu, click on **File,** then **Open TRAY**.

14.4.2 Click twice on the name of the TRAY y you wish to open. (**Phenols.tra**) The first rows of the TRAY spreadsheet are blue. This denotes that these samples are actually

Calibration Standards. This is from the method and will not change. The Sample **Type** is **Cal Std.** And the **Level** is NEVER 0; it will be a number form 1 to 14 (This will match the level in the Analyte Table in the method.) The Cup Number (**Cup #**) refers to the cup number in the Standard Rack and will be a number between 1 and 14.

14.4.3 Edit the **Sample ID.** Using the mouse, move the pointer to the cell and click once; type in the new ID. In the Sample Rack Loading Aid (Top Left) the sample cup you are editing will be in green.)

14.4.4 The Cup Numbers (**Cup #**) for sample will be anything between 1 to 90.

14.4.5 The **Level** column is **0** for all samples of **Sample Type** of **unknown**

14.4.6 The **Reps** for samples and standards is **1.**

14.4.7 To Schedule manual QC See Appendix C (DQM Plan)

14.5 Click on the **Run TRAY** button. (Or from the Main Menu, click on **TRAY** then **Run TRAY.**)

14.5.1 Leave the Method and TRAY boxes empty to use the one that are active (open).

14.5.2 In the Data File box enter the name for your run, (i.e. 981006Ca. YYMMDD C (for Calibration) and a (for number of the run... a b c...)). The extension *.fdt (FIA Data) is used by default.

14.5.3 The Autosampler Position refers to the autosampler sample rack position: 1, 2 or 3.

14.5.4 **Skip Recalibration Block** Box can be checked when you want to run a TRAY that contains Cal standards and samples but you want only want to run samples

14.5.5 While the TRAY is processing, you can view the peaks and runtime report.

14.5.6 If the baseline does not appear on the screen change Display options.

14.5.7 Click **Method**, then **Display Options**

14.5.8 Specify the channel: 1.

14.5.9 Specify the voltage scale on the Y-axis (i.e. -1 to 3).

14.5.10 Click **OK.**

14.6 Analyze samples for Phenols. Click on the **RUN** button. The TRAY will start running. After the pump is allowed to pump at a normal speed for the period specified it the method, pump timing, the autosampler will move to the first calibration standard. The Standards and samples are done in Row order no matter where they are located on the sampler.

14.7 While running you will see a **STOP** sign on the tool bar, to suspend the TRAY click on the STOP sign. The TRAY will pause and the computer gives you the choices of aborting or resuming the TRAY. At the end of the TRAY, after the last sample ID entered is reported, the sample probe should return to the wash bath and the **STOP** button will turn back into the **RUN** button.

14.8 System Shutdown Procedure

14.8.1 Remove the regent transmission lines and place into the rinse solution and pump for 5 minutes at standard speed.

14.8.2 Place the lines into reagent water and allow the system to rinse 5 to 10 minutes at standard speed.

14.8.3 Remove the lines from the reagent water and allow all liquid to be pumped out of the manifold.

14.8.4 Turn off the pump and release the pump tube cartridges tension. Press the tube cartridges holders on the sides of the Ismatec pump.
14.8.5 Turn off the Header by Reducing the Set Point to a temperature lower than room temperature (e.g. 15°C).
14.8.6 Close all files
14.8.7 Switch off the master power strip

14.9 Documentation requirements. Record the following information in the appropriate logbook or data file. Include any deviations from this procedure.

Analyst initials, date [and time if required by the specific project or QAPP] of analysis, sample number or ID, initial sample volume or weight processed, final digestate volume, calibration standard sample or solution identifier, QC sample or solution identifier, reagent solutions identifiers, any dilution information, [beginning and ending times of analytical steps if required by the specific project or QAPP], data file name or batch ID, instrument method name, visual observations, and any other information as deemed necessary.

14.10 Routine Maintenance – Record all maintenance in the logbook. See Appendix F

15.0 Data Reduction, Calculations and Loading

15.1 The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

NOTE: The LIMS program will convert µg/L concentrations from the instrument readout to mg/L or mg/Kg (part per million).

15.2 Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard must be diluted and reanalyzed.

15.3 Aqueous Samples: The concentration readout for aqueous sample distillates is µg phenol/L. It does not need further data reduction unless the initial sample volume was less than 50 mL. If less than 50 mL sample was distilled, calculate the concentration as follows:

Concentration in mg phenol /L = readout * 0.001 (50 mL/ initial sample volume analyzed in mL)

15.4 For sample results greater than the highest calibration standard, dilute the sample in a 10 mL centrifuge tube using reagent water. Pipet in the appropriate volume of sample into the tube, dilute to the mark with reagent water, cap and mix well. Record the volume of distillate used for analysis. Dilution Factor = (10 mL/ distillate volume analyzed in mL). Use the dilution factor and calculate the concentration in aqueous samples as follows:

Concentration in mg phenol /L = readout * 0.001 (10 mL/ distillate volume analyzed in mL)

15.5 Soil Samples: The concentration readout for soil samples must be multiplied by the following factor: Factor = (50 / sample weight in g). Calculate the concentration in soil samples as follows:

Concentration in mg phenol /Kg = readout * 0.001 (50/ sample weight in g)

For soil sample results greater than the highest calibration standard, follow the dilution procedure in section 15.4

15.6 Soil samples reported on a dry weight basis: The concentration is divided by the decimal equivalent of the percent residue of the soil at 105^0C.

15.7 Report results in mg phenol/L, or mg phenol/Kg.

15.8 The procedure for uploading data into the LIMS system is detailed in SOP 1400 LIMS.

15.9 See SOP 001 Quality Assurance Manual, sections 5.4 and 5.5 for additional calculations of precision and accuracy.

16.0 Method Performance

Demonstration of Capability (DOC)

All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

A quality control (QC) reference concentrate is required containing phenols at a concentration of 0.02 mg/L for aqueous samples and 1 mg/Kg for soil samples using the ICV/LCS solution. The QC reference sample is made using stock standards prepared independently from those used for calibration.

For each analyte calculate the mean recovery (x) and standard deviation (s) and the average % Recovery (%R). Compare x and s and %R with the corresponding acceptance criteria for accuracy and precision, respectively. Note: For aqueous samples, x must be within 0.02 ± 0.004 mg/L and s must be less than 0.004 mg/L and %R must be within 100 ± 20%. Note: For soil samples, x must be within 1.0 ± 0.2 mg/Kg and s must be less than 0.2 mg/Kg and %R must be within 100 ± 20%. These limits are taken from established in-house criteria If x and s and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If x or %R falls outside the range for accuracy, or s exceeds the precision limit, then the system performance is unacceptable for that analyte and corrective action must be taken.

Comparison to Reference Method Data

There are no stated reference method criteria for ICV, LCS, Duplicate Sample or MS/MSD recoveries in EPA Method 9066. Verify the calibration with an independent check sample every 15 samples. The duplicate should be performed every 10 samples.

EPA method 9066 provides performance data for reproducibility for phenol analysis in sewage samples at several concentrations. Standard deviations range from 13% RSD for a sample concentration of 0.0038 mg/L to 1% RSD for a sample concentration of 0.089 mg/L.

EPA method 9066 provides performance data for phenol spike recoveries in sewage samples at several concentrations. Recovery of the spike ranges from 78% for a sample concentration of 0.0053 mg/L to 98% for a sample concentration of 0.082 mg/L.

In-House Control Limits

Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data is compared to the specifications of the reference method. If the in-house limits are within the specifications of the reference method, the control limits are updated in LIMS. If the in-house limits are not within specifications, an investigation is performed to determine the cause(s) of the problem and a corrective action is completed. The analysis may continue until enough data points are collected to regenerate new control limits. Any QC data generated outside of reference method limits during that time frame is flagged.

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained. After the analysis of 20 laboratory control samples and surrogates, calculate the average percent recovery (R) and the standard deviation of the percent recovery (S).

Control limits for the method parameters are generated by the QC staff and distributed to the analysts via updates to the LIMS control charts. The control limits are calculated based on in-house performance data. At a minimum, these limits are updated annually.

17.0 Pollution Prevention

The preparation of excessive volumes of laboratory reagents and standards shall be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards shall be disposed in compliance with the laboratory waste disposal program and applicable waste disposal regulations. With the consent of the client, the samples may be returned to their origin for treatment.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory staff are required to protect the laboratory's and our clients' business information when disposing of recycling or waste from the facility.

18.0 Data Assessment and Criteria for Quality Control Measures

The laboratory must maintain records to document the quality of data that is generated. Ongoing quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. The data review is conducted according to SOP 1250 Data Review.

Method Blank (MB)
If the blank exceeds the RL (the lowest calibration standard), the source of contamination must be investigated and corrective actions taken.

Affected samples must be reprocessed and reanalyzed **or** Data must be appropriately qualified if:

1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the SOP or by regulation, AND is greater than 1/10 of the amount measured in any sample.

2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

Laboratory Control Sample (LCS)
The results of the individual batch LCS are calculated in percent recovery (%R) and compared to established acceptance criteria (in-house limits). LCS %R limits are 100 ± 20%. If the LCS is outside the acceptance criteria, the analytical system is "out of control". Any affected samples associated with an out of control LCS must be reprocessed and reanalyzed or the results reported with appropriate data qualifiers.

Matrix Spikes
The results from MS/MSD are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). See the STAT QAM, Section 5.4 for the calculation for RPD. Results are compared to established acceptance criteria (in-house limits). For aqueous samples, MS/MSD %R limits are 100 ± 25% and RPD limits are 20%. For soil samples, MS/MSD %R limits are 100 ± 25% and RPD limits are 20%. For matrix spike results outside established criteria corrective action must be documented, or the data for the spiked sample is reported with appropriate data qualifying codes.

Duplicates
The results from laboratory Duplicates are designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). See the STAT QAM, Section 5.4 for the calculation for RPD. Results are compared to established acceptance criteria (in-house limits). RPD limits are 20%. For duplicates results outside established criteria corrective action must be documented, or the data for the duplicate sample is reported with appropriate data qualifying codes.

19.0 Corrective Actions for Out-of-Control Data

The process for handling corrective actions is found in SOP 230 Corrective Action.

If the CCV, MB, LCS/LCSD, MS/MSD, or lab duplicate recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the samples is suspect and is only reported for regulatory compliance purposes with the appropriate corrective action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. Final data results must be qualified in the client report for reported results not meeting the laboratory-defined criteria.

1) Review standards preparation logbooks. Check all calculations and ensure dilution factors are properly recorded.
2) Re-prepare the suspected standard or QC sample to identify possible preparation errors of the standard or QC sample.
3) Re-Analyze the samples when the CCV or LCS is not within acceptable limits.
4) Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

20.0 Contingencies for Handling Out-of-Control Or Unacceptable Data

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discusses options with the client. Reanalysis or reporting the data with qualification are alternatives. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.

The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

21.0 Waste Management

The STAT Analysis Corporation SOP 1130 Waste Disposal identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded accordance with SOP 1130 Waste Disposal.

22.0 References

22.1 Method 9066, U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" Update III, December 1996

22.2 National Environmental Laboratory Accreditation Conference (NELAC), Current version at date of signing, USEPA Office of Research and Development, Washington, DC EPA600/R-99-068
22.3 Determination of Total Recoverable Phenols by Flow Injection Analysis. QuikChem Method 10-210-001-A
22.4 STAT Analysis Corporation Quality Assurance Manual
22.5 STAT SOP 003 Chemical Hygiene Plan
22.6 STAT SOP 230 Corrective Action
22.1 STAT SOP 1000 Control and Use of Laboratory Notebooks
22.2 STAT SOP 1010 Analytical Standards and Reagents Receipt and Preparation
22.3 STAT SOP 1020 Laboratory Glassware Cleaning
22.7 STAT SOP 1040 General Laboratory Practices
22.8 STAT SOP 1130 Waste Disposal
22.9 STAT SOP 1210 Method Detection Limits
22.10 STAT SOP 1230 Training
22.11 STAT SOP 1250 Data Review
22.12 STAT SOP 1400 LIMS
22.13 STAT SOP 3620 Phenols Distillation by EPA 9065
22.14 QuikChem 8000 Automated Ion Analyzer Omnion FIA Software.
22.15 QuikChem 8000 Automated Ion Analyzer Continuum Series "Flow Injection Analyzer" Hardware Installation and System Operation.

23.0 Forms, Figures, Tables, Diagrams, Flowcharts, Attachments or Validation Data

23.1	Appendix A	Troubleshooting
23.2	Appendix B	Creating a Method
23.3	Appendix C	DQM Plan
23.4	Appendix D	Manifold Diagram
23.5	Appendix E	Manifold Installation/ Removal
23.6	Appendix F	Maintenance Schedule

Appendix A – Trouble Shooting

Keep all modules clean and dry at all times.

Keep in Stock:	Pump tubes	green
		red
		red
	Teflon tubing:	0.8 mm id Manifold
		0.5 mm id Low Flow Manifold
		0.6 mm id Restriction Coil
		o-rings
		Transmission Tubing

Routine Maintenance (See Appendix F for the Maintenance Schedule)

Pump

- After each day rinse the cartridges in DI Water to wash any spills. Clean pump surfaces, except rollers, with a wet cloth. Dry all surfaces.
- If rusty clean rollers with steel wool. A light coat of silicon spray can be applied by spraying on a lint free cloth and holding it to the moving rollers.
- Check for wear, cracks or acid damage on the cartridges ad holders.
- Replace pump tubes that start to show signs of wear.
- If the pump tubes burst, clean all cartridges and holders, as well as, the pump, immediately.
- All surfaces should be kept clean. Use a damp cloth to clean the module surfaces. Dry surfaces thoroughly.

Valve Modules

- Keep the instrument clean and dry at all times.
- Replace o-rings as necessary
- When changing the o-rings, clean the valve ports with cotton swab and reagent water. This will help remove any dirt or precipitate that may prevent a good seal. If a leak persists, replace the new o-rings and make sure the connector itself does not have precipitate on the thread.
- All surfaces should be kept clean. Use a damp cloth to clean the module surfaces. Dry all surfaces thoroughly.
- The internal sample loop valve will give thousands of injections without trouble. The rotor seal wears with use and is the only part that needs routine replacement.
- Detector Modules and Flow Cell
- If a flow cell appears to leak remove it immediately to keep all liquid form the electronics inside the detector head.

Instrument Troubleshooting

For problems with the instrument see the QuikChem 8000 Automated Ion Analyzer Continuum Series "Flow Injection Analyzer" Hardware Installation and System Operation Manual under the Troubleshooting Section.

Appendix B – Creating A Method

1.1 Click on the **Method** button, or from the Main Menu click on **File**, then **New Method**.
1.2 Then Method will open and you will see the Analyte Table
1.3 Click on Analyte Name of channel and press backspace to delete. Enter the new analyte name. If an analyte name is not present the system will ignore that channel.
1.4 Fill out the analyte Table.

Channel	1
Analyte Name	Phenol
Concentration	µg/L
Level 1	200
Level 2	100
Level 3	50
Level 4	10
Level 5	5.0
Level 6	0.0
Calibration Rep Handling	Average
Calibration Fit Type	1st Order Polynomial
Force Through Zero	No
Weighting Method	None
Concentration Scaling	None
Chemistry	Direct
Injection to Start Peak	20s
Peak Base Width	29s
% Width Tolerance	100%
Threshold	2000

1.5 From the Main Menu, click on **Method**, then **Valve Timing**
1.6 Enter the **Method Cycle Period** 40s
1.7 Sample Reaches the first valve 18s (For Standard Pump Sample Assembly) (travel time from sample probe to port 6 usually 24 s if dilutor enter 28s)
1.8 **Load Period** 20s
Load Time 0s
Inject Period 20s
Notice that Load Period + Injection Period = Cycle Period
1.9 Click **OK**

1.10 From the Main Menu, click on **Method**, then **Sampler Timing**.
1.11 The Sample Prep Sequence box is optional
1.12 Enter the **Minimum Probe in Wash Period** 5.0 s
1.13 Enter the **Probe in Sample Period** 34s
1.14 Click **OK**.
1.15 From the Main Menu, click on Method, then Pump Timing

Appendix B – Creating a Method (cont.'d)

1.16 Check the **Go to Standby on Idle** box X
1.17 Enter **Idle Before Standby** 180.0s
1.18 Enter **At Speed Before Analysis** 45.0s
1.19 Click **OK.**
1.20 From the Main Menu, click on **File**, then **Save Method As**
1.21 The FIA Method File Header will appear. Write a method description (analyte and sample loop size) and click **OK** (i.e. **Phenols Sample Loop XXX cm**).
1.22 The method "Save As" dialog box will appear.
1.23 C:\ Omnion\Methods\
Change the name to **Phenols.met**

II To Fine Tune the Method

1.0 After you have run a data.
- 1.1 Open a file from the main menu click on File, Open Data and click twice on the Data file name (e.g. 981006 Ca.fdt.).
- 1.2 Load the original method form this data by clicking on the Data menu the Load Method.
- 1.3 You should see the peaks on the screen.
- 1.4 Click on Data then Reanalyze Data. (Or click on the Reanalyze button.)

2.0 Fine Tuning the Threshold
- 2.1 Look before and after the TRAY to see some baseline.
- 2.2 From the menu click on **Method**, then **Graphical Events Programming...**
- 2.3 Click on **Threshold.**
- 2.4 The Status bar will prompt you to "Select Start of Baseline Section".
- 2.5 Set the cursor at the beginning of the baseline region and click once.
- 2.6 You will be prompted to "Select End of Baseline Section".
- 2.7 Move the mouse pointer to the end of the baseline region and click again.
- 2.8 Omnion calculates a Threshold value.
- 2.9 Click on **OK** to enter the value in the Analyte Table of your method

3.0 Fine Tuning the Peak Base Width
- 3.1 From the menu click on **Method**, then **Graphical Events Programming...**
- 3.2 Click on **Peak Base Width.**
- 3.3 You will be prompted to "Select Start of Peak"
- 3.4 Set the cursor at the beginning of the high standard and click once.
- 3.5 You will be prompted to "Select End of Peak"
- 3.6 Move the cursor to the end of the peak and click again.
- 3.7 Omnion calculates a Peak Base Width.
- 3.8 Click on **OK** to enter the value in the Analyte Table of your method

Appendix B – Creating a Method (cont.'d)

4.0 Save these new method parameters by clicking on **File**, then **Save Method As**.

5.0 Viewing a Method's Calibration

5.1 Up to 4 replicates of each Standard can be applied to a method's calibration.

5.2 If not already open, load the method form the data file by clicking on Data, Load Method.

5.3 Click on the button, (or from the main menu click on **Method**, then **Review Analyte Calibration Curve**. The Review Analyte Calibration window will appear.

5.4 Click **Fit**, and then **Clear**, to 'Clear' the Calibration Replicate Table.

5.5 Click **Exit**.

5.6 Click the **Analyze** button. The re-analysis occurs exactly as it did in the actual TRAY run.

5.7 Click on the **Review Calibration** button again to see the curve.

6.0 Editing the Calibration

6.1 Clicking twice on any of the results in the calibration replicate Table will turn it red and make it unused in the calibration.

6.2 To Use the point again, click twice on it again, and it will turn form red back to blue.

Appendix C – DQM Plan

DQM (Data Quality Management) Plan
Consists of one or several DQM Sets. Each set has one or more samples. The DQM has 3 sections; the DQM Set box, the DQM Sample Box, and the Channel Data box.

1.0 Adding DQM Sets
- 1.1 Click TRAY, then Load DQM.
- 1.2 In the DQM Set Box click on the drop-down button showing the **DQM Set ID**, type in a new name. (Check Standards, Duplicates, Matrix Spikes)
- 1.3 For the Check Standards Set check the **Automatic** box **X**.
- 1.4 Click **ADD** to add the set.

NOTE: Automatic Sets never have their sample info in the TRAY table and the samples are loaded in the standards rack. Manual sets have their sample info in the TRAY table and the samples are loaded in the sample rack.

2.0 Adding DQM Samples
- 2.1 In the DQM Sample Box, click on the drop-down button showing the **DQM Sample ID** and type in a new name (ICB, ICV, CCV, CCB, Method Blank, Dup 1, Spike, Spike Dup)
- 2.2 Click on the **Append** button.
- 2.3 **Replicates** should bel or **2.**
- 2.4 Click on the drop-down button showing the **Type** and select the type of sample. (Blank, Unspiked, Spiked, AbsChkStd, RelChkStd, Dup 1, Dup 2)
- 2.5 For the Automatic Check Samples enter the **Standard Rack Cup.**

3.0 Editing Channel Data information.
- 3.1 In the Channel Data Box, select the appropriate channel.
- 3.2 If you can't see the Test 1 row, click on the **Add Test** button.
- 3.3 The Test Explanation spells out the test that will be done for this DQM Sample and depends on what Type the DQM Sample is.
- 3.4 If the standard has a known concentration it needs to be entered in the **Known Conc** box along with the **Conc Units.**
- 3.5 Enter the **Test Limit.** 10.000% difference.
- 3.6 Select a Fail Action from the drop-down menu. **Recalibrate & Repeat, Alarm & message.**
- 3.7 Enter the Pass/ Fail Message.
  NOTE:A Test Passes if test value < = Test Limit
  A Test Fails if Test value > Test Limit
- 3.8 You can perform more then 1 test on each check sample.

4.0 Scheduling Automatic DQM Sets
- 4.1 From the Main Menu, click on **TRAY**, then **Auto DQM Schedule**

Appendix C – DQM Plan (cont.'d)

4.2 Select the **Auto DQM Set** from the drop-down menu.
4.3 Check the box(es) for the frequency of the sample
4.4 Click **OK.**

5.0 Inserting Manual DQM Sets
5.1 Click on the **TRAY Table** button.
5.2 Click on the row number before which you want to insert the check sample.
5.3 From the Main Menu, click on **TRAY**, then **Manual DQM Insertion.**
5.4 Click on the **Manual DQM Set** you wish to insert.
5.5 Enter the **Sample ID**'s of the DQM samples to reflect the actual identity of the samples. (Note: The manual DQM sample rows are green.)
5.6 Renumber the Cup Numbers (**Cup #**) below the inserted Manual DQM Set by clicking and dragging all the rows and cup numbers you wish to renumber.
5.7 Click on **TRAY**, then **Renumber Cups** (or **Ctrl-R**)
5.8 Enter the **Starting Number**
5.9 Enter an **Increment** of **1.**
5.10 Then click **OK.**

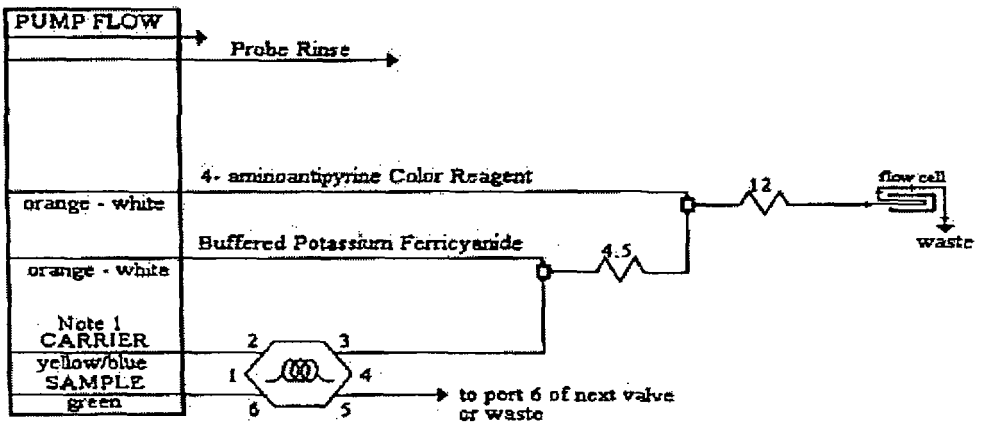
6.0 Calibration Pass/Fail Criteria
6.1 From the Main Menu, click on Method, then Calibration Failure Criteria
6.2 Select the appropriate channel.
6.3 Check the Minimum Correlation Coefficient (R2) Box and enter 0.995
6.4 Check the Maximum % Residual All Levels Box, and enter 10.0.
6.5 If the calibration passes, a message will be reported and the tray will continue to the nest row.
6.6 If the calibration fails, a fail message will be reported and you will be given a choice to Abort or Continue the tray.

7.0 Save the DQM Plan
7.1 From the Main Menu, click File, then Save DQM Plan, or Save DQM Plan As... and enter the File name PHENOL.dqm. The default extension is *.dqm.

Appendix D – Manifold Diagram

17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1. PHENOLICS MANIFOLD DIAGRAM:



Sample loop = 150 cm
QC8000 sample loop = 155.5 cm

Interference Filter = 500 nm

Manifold tubing is **0.8 mm (0.032 in)** i.d. This is 5.2 uL/cm.

CARRIER is helium degassed water.

4.5 is **70.0** cm of tubing on a 4.5 cm coil support

12 is **255** cm of tubing on a 12 cm coil support

APPARATUS: Standard valve, flow cell, and detector head modules are used.

Note 1: **Carrier Line** for the AE: use a green/green pump tube.

Note 2: Transmission tubing should be replaced with 1 m of Teflon manifold tubing (0.8mm i.d.) as transmission tubing may contain leachable phenolics. Use Teflon tube connectors (Part# 50008) with PTA as line weights with pin removed.

Appendix E – Manifold Installation/Removal

1.0 Unwrap the transmission lines from around the manifold and place the manifold over the sample-processing module.

1.1 Remove the transmission lines from the union on the 650 cm side of the heating unit, insert both ends through the hole in the manifold and reseat the manifold onto the sample-processing module.

1.2 Make the Injection Valve Connections as follows:

1.3 Port 1 & 4 The Sample Loop 0.8 mm i.d. length 40 cm.

1.4 Port 2 Carrier line is connected to Port 2 -> from valve, disconnect from fitting and connect to port 2 0.8 mm id and 30 cm long.

1.5 Port 3 20 cm 0.8 mm id between port 3 and the fitting on the manifold next to the label -> from valve where the carrier line was connected.

1.5 Port 5 15 cm 0.8 mm id. between port 5 and the waste line.

1.6 Port 6 Sample Line. 130 cm (Varies method to method) connected to the probe on the autosampler, pump tube adapter, and 20 cm tubing connected to port 6 of valve.

2.0 Flow Cell Top tubing connected to waste line. Bottom line connected to the fitting in the manifold next to the label to flow cell ->.
Attach one of the lines from the heater to the union going to the flow cell.
Attach the other heater line to the pyridine-Barb. Acid Tee fitting.

3.0 Pump

3.1 Sample Line - Autosampler to injection valve 6

3.2 Wash Line - Reagent water to wash reservoir on the autosampler

3.3 Reagents Lines- Varies from method to method.

3.4 On the Ismatec Pump Cartridges the arrows point towards the System Unit with the tension lever on the left.

3.5 Place all of the reagents lines into corresponding containers or Reagent water.

3.6 Move tension levers to the maximum tension (Top Far Right Position)

3.7 Clamp down all pump tube cartridges. (Press down one side at a time.)

3.8 Move tension lever back from the top far right until it makes a clicking sound.

3.9 Set the reagent pump speed to 35.

3.10 Turn on the pump.

3.11 Depress the green button to turn the pump ON. The System Unit will take control over the pump speed. (The yellow button is the override standby button.)

3.12 Check to confirm that the probe wash reservoir is filing with rinse water.

3.13 Check for Leaks on the manifold, valve, flow cell or any of the connections.

3.14 Do NOT leave any pump tubes clamped down when the pump is shut off for more then a few minutes.

3.15 To Remove cartridges, Press the sides of the pump holder on which the cartridge is engaged.

Appendix E – Manifold Installation/Removal (cont.'d)

4.0 Insert the interface filter into the detector module.

II Manifold Removal Procedure

1.0 Rinse the manifold

1.1 Detach the manifold tubing from the manifold fitting that is connected to Port 3 at the injection valve. Leave the piece of tubing attached to the injection valve.

1.2 Disconnect the carrier pump tube from Port 2 of the injection valve. Take this tubing and connect it to the manifold fitting that was connected to Port 3.

1.3 Detach output of the manifold from union on the flow cell tubing leave the union connected to the flow cell.

1.4 Remove the backpressure loop, if necessary.

1.5 Detach heating unit tubing from the manifold, and reconnect to the union, underneath the manifold (650 cm side).

1.6 Remove all manifold pump tubes from cartridges.

1.7 Remove the interface filter from the detector module.

1.8 Remove the sample loop from Port 1 & 4 valve.

1.9 Remove manifold from the Sample Processing Module (Channel).

1.10 Carefully wrap the transmission lines around the manifold and store it in the plastic bubble bag.

Appendix F – Recommended Maintenance Schedule

All listed maintenance is performed as needed.

Daily	
AutoSampler	Clean Surfaces
AutoDilutor	Clean Surfaces
	Prime dilutor with DI water after using any other diluent
Pump	Clean Surfaces
	Rinse cartridges
Detector	Dry and Clean all Surfaces
System Unit	Keep Dry and Clean
Weekly	
Injection Valves	Clean Ports and valve connections
Monthly	
Autosampler	Clean rods/ moving parts
Pump	Spray silicon on cloth and rub on rollers
	Replace pump tubes
	Clean pump tube adapters
Manifold	Clean union and tee
6 Months	
Injection valves	Replace o-rings
Manifold	Replace o-rings
Yearly	
Manifold	Replace all tubing
Flow Cells	Replace flares and o-rings
Computer	Clean hard drive

Attachment 2

Example Field Forms For Analytical Samples

FedEx. US Airbill
Express

FedEx Tracking Number 8683 6326 5688

0200 Sender's Copy

1 From *Please print and press hard.*

Date 12/18/08 Sender's FedEx Account Number S**********

Sender's Name Tim Gilles Phone 630 878-1780

Company Burns & McDonnell

Address 100 E. Seahorse Drive

Dept./Floor/Suite/Room

City Waukegan State IL ZIP 60085

2 Your Internal Billing Reference First 24 characters will appear on invoice. 49452-9524

3 To

Recipient's Name Sample Receiving Phone (847) 967-6666

Company Environmental Monitoring & Technologies

Recipient's Address 8100 N. Austin Avenue

We cannot deliver to P.O. boxes or P.O. ZIP codes.

Dept./Floor/Suite/Room

Address

To request a package be held at a specific FedEx location, print FedEx address here.

City Morton Grove State IL ZIP 60053

4a Express Package Service *Packages up to 150 lbs.*

- [X] FedEx Priority Overnight — Next business morning.* Friday shipments will be delivered on Monday unless SATURDAY Delivery is selected.
- [ ] FedEx Standard Overnight — Next business afternoon.* Saturday Delivery NOT available.
- [ ] FedEx First Overnight — Earliest next business morning delivery to select locations.* Saturday Delivery NOT available.
- [ ] FedEx 2Day — Second business day.* Thursday shipments will be delivered on Monday unless SATURDAY Delivery is selected.
- [ ] FedEx Express Saver — Third business day.* Saturday Delivery NOT available.

FedEx Envelope rate not available. Minimum charge: One-pound rate. * To most locations.

4b Express Freight Service *Packages over 150 lbs.*

- [ ] FedEx 1Day Freight* — Next business day.** Friday shipments will be delivered on Monday unless SATURDAY Delivery is selected.
- [ ] FedEx 2Day Freight — Second business day.** Thursday shipments will be delivered on Monday unless SATURDAY Delivery is selected.
- [ ] FedEx 3Day Freight — Third business day.** Saturday Delivery NOT available.

* Call for Confirmation; ** To most locations.

5 Packaging

- [ ] FedEx Envelope*
- [ ] FedEx Pak* — Includes FedEx Small Pak, FedEx Large Pak, and FedEx Sturdy Pak.
- [ ] FedEx Box
- [ ] FedEx Tube
- [X] Other — * Declared value limit $500.

6 Special Handling — Include FedEx address in Section 3.

- [ ] SATURDAY Delivery — NOT Available for FedEx Standard Overnight, FedEx First Overnight, FedEx Express Saver, or FedEx 3Day Freight.
- [ ] HOLD Weekday at FedEx Location — NOT Available for FedEx First Overnight.
- [ ] HOLD Saturday at FedEx Location — Available ONLY for FedEx Priority Overnight and FedEx 2Day to select locations.

Does this shipment contain dangerous goods? One box must be checked.

- [X] No
- [ ] Yes — As per attached Shipper's Declaration.
- [ ] Yes — Shipper's Declaration not required.
- [ ] Dry Ice — Dry ice, 9, UN 1845 ____ x ____ kg

Dangerous goods (including dry ice) cannot be shipped in FedEx packaging.

- [ ] Cargo Aircraft Only

7 Payment *Bill to:* Enter FedEx Acct. No. or Credit Card No. below.

- [ ] Sender — Acct. No. in Section 1 will be billed.
- [ ] Recipient
- [X] Third Party
- [ ] Credit Card
- [ ] Cash/Check

FedEx Acct. No. / Credit Card No. S********** Exp. Date

Total Packages	Total Weight	Total Declared Value†
1	40	$ .00

†Our liability is limited to $100 unless you declare a higher value. See back for details. By using this Airbill you agree to the service conditions on the back of this Airbill and in the current FedEx Service Guide, including terms that limit our liability.

8 Residential Delivery Signature Options If you require a signature, check Direct or Indirect.

- [ ] No Signature Required — Package may be left without obtaining a signature for delivery.
- [ ] Direct Signature — Someone at recipient's address may sign for delivery. Fee applies.
- [ ] Indirect Signature — If no one is available at recipient's address, someone at a neighboring address may sign for delivery. Fee applies.

520

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RETAIN THIS COPY FOR YOUR RECORDS.

Example Airbill for Sample Shipping
(if laboratory courier not used)

Client: WMGCP Site Performing Settling Defendants OU#2
Location: Waukegan Manufactured Gas & Coke Plant Site

Sample ID: E-01-1-081028-001
Collection Date: 10/28/08 Collection Time: ___:___
Collected By: T. Gilles

Bottle Type: 500 mL Plastic Preservative: HNO_3
Analysis: Arsenic

Sample Label

Burns & McDonnell
1431 Opus Place, Ste. 400
Downers Grove, IL 60515

Signature _____

Date _____ # **-103254**

Custody Seal

Burns & McDonnell
SINCE 1898

Request for Chemical Analysis and Chain of Custody Record

Burns & McDonnell Engineering
1431 Opus Place
Downers Grove, Illinois 60515
Phone: (630) 724-3200 Fax: (630) 724-3201

Attention:

Laboratory:

Address:

City/State/ZIP:

Telephone:

Document Control No.:

Lab. Reference No. or Episode No.:

Project Number:

Sample Type

Site Name:

Matrix

Sample Number			Sample Event		Sample Depth (in feet)		Sample Collected		Matrix			Number of Containers	Parameter/Method Code							
Group or SWMU Name	Sample Point	Sample Designator	Round	Year	From	To	Date	Time	Liquid	Solid	Gas									Remarks

Sampler *(signature)*:

Sampler *(signature)*:

Custody Seal Number

Special Instructions:

Relinquished By *(signature)*:
1.

Date/Time

Received By *(signature)*:

Date/Time

Ice Present in Container:
Yes ☐ No ☐

Temperature Upon Receipt:

Relinquished By *(signature)*:
2.

Date/Time

Received By *(signature)*:

Date/Time

Laboratory Comments: